Double Beam
Atomic Absorption Spectrophotometer

User Manual and Maintenance Guide

Zeeman Double Beam
Atomic Absorption Spectrophotometer
TL-1800AA Series

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Version 1806

Contents

Chapter 1 Introduction

1.1 Instrument Description

AAS is a multifunctional Atomic Absorption Spectrometer with background correction methods, D_2 lamp background elimination, Self-absorption and Zeeman background deduction. It is a high precision analytical instrument integrates flame atomic absorption spectrometry (abbreviated as FAAS), and hydride generation atomic absorption spectrometry (abbreviated as HG-AAS), and graphite furnace atomic absorption spectrometry (abbreviated as GFAAS), and atomic emission spectrometry (abbreviated as AES). An auto-sampler is optional to equip with graphite furnace atomic absorption spectrometry. AAS is widely used in geology, metallurgy, mining, petroleum, chemical industry, mechanical, agriculture, food industry, medicine, health, antiepidemic, environmental protection, and other industries. And it is an essential high precision analytical instrument for scientific research institutes, colleges and universities, and related enterprises.

AAS can be used for analyzing metallic elements, amphoteric elements, and alkali elements. It’s a high precision analytical instrument with high performance and its determination range can be from ppb to ppt. Following are the features of AAS:

- ✧ Advanced determination of flame atomic absorption spectrometry

  It is developed by experienced professors and senior engineers who are engaged in analytical instrument industry of atomic absorption spectrometry for many years. The background correction with Deuterium hollow cathode lamp can eliminate the interference of molecular absorption in low content determination. The detection sensitivity is greatly improved, and the value is less than 0.015 μg/ml/1%.
- ✧ High detection sensitivity and low detection limit of hydride generation atomic absorption spectrometry

  The import known-how such as the advanced pneumatic component theory, and high performance hollow cathode lamp guarantees the hydride generation atomic absorption spectrometry performing a high detection sensitivity and low detection limit. For example, the detection sensitivity of Arsenic element is better than 0.08 ng/ml/1%. The instrument precision is also greatly improved.
- ✧ Convenient software operation system provides reliable data processing result

  The data processing system of AAS is an application of new data processing achievement in the field of atomic absorption spectrometry. It provides reliable data processing result. Meanwhile, the special designed operation software system is advanced and convenient.
- ✧ A partly substitution of graphite furnace atomic spectrometry

  The atomic absorption spectrometry together with on-line enrichment by the pneumatic flow injection system designed by ourselves can determine some elements and obtain a high sensitivity, that means it is a partly substitution of graphite furnace atomic spectrometry for certain elements. Thus, it not only reduces the testing cost, but also improves the instrument utilization.

- ✧ Special designed double-loop power supply for the high performance hollow cathode lamp

  AAS uses special designed double-loop power supply for the high performance hollow cathode lamp, which is convenient for lamp adjusting. It not only improves the lamp energy of some element, but also improves the detection sensitivity to several times. And the performance indexes of the instrument such as stability and detection sensitivity are greatly improved.

- ✧ Unique micro-volume sampler with constant current pulse

  A unique micro-volume sampler with constant current pulse that designed based on the pneumatic component theory is optional to equip with flame atomic absorption spectrometry. The sample volume can be reduced to 0.1 ml, however, there is few influence on the detection sensitivity. And a relative standard deviation about of 1% can be obtained. Thus, it is very significant to medicine and health care industry.

- ✧ Innovative detection system

  Innovative atomic absorption detection system guarantees the instrument having an excellent stability.

- ✧ USB communication port

  The instrument is controlled by the USB port of computer, which is an advanced communication method. User can choose the computer as required, and that is also available for other laboratory use. However, to avoid viruses, please don’t use this computer for internet surfing.

1.2 Instrument Structures

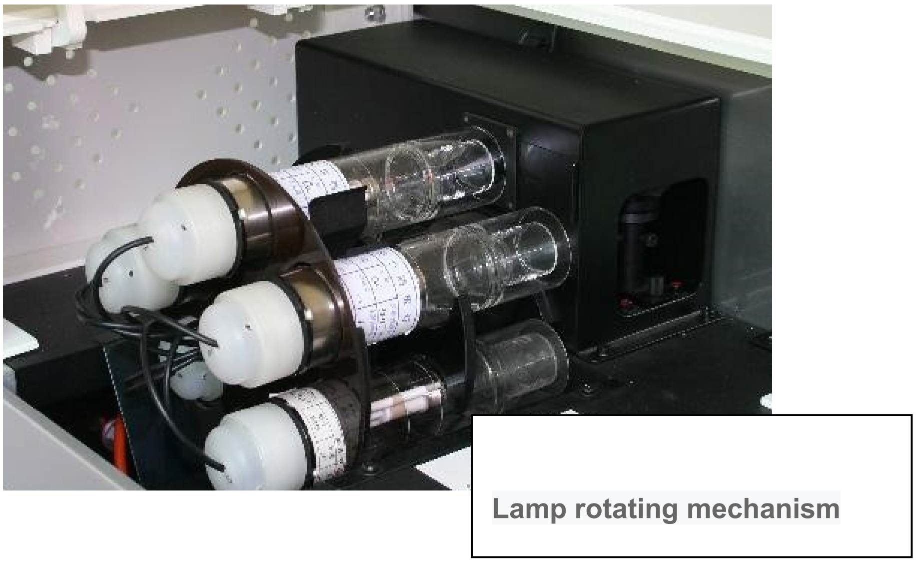
AAS multifunctional atomic absorption spectrometer includes following parts: Atomic absorption spectrometer together with graphite furnace and flame atomization system, power supply of graphite furnace, auto-sampler (optional), computer and printer, and air compressor.

1. The main structure of the atomic absorption spectrometer

   The main structure of the atomic absorption spectrometer includes the light source and optical system, the gas control system, security control system, graphite furnace and flame atomization system, electronic control and detection system, mechanical lifting system, and other optional systems.

   1) The light source system

      The light source system is composed of a light rack and a deuterium lamp holder. The light rack can hold six lamps, and the deuterium lamp holder is only for the deuterium hollow cathode lamp.



Lamp rotating mechanism

Among the six positions of the light rack, four positions (lamp holders from No. 1 to No. 4) are for normal hollow cathode lamps (abbreviated as HCL), two positions (lamp holders of No.5 and No. 6) are for high-performance hollow cathode lamps (abbreviated as HPHCL).

For HCL, each lamp position can be adjusted automatically, and the lamp current can be set from 0.5 mA to 9.9 mA. Usually, the best working current is between 1 mA and 5 mA. The system default value is 3 mA.

For HPHCL, it is controlled by the main current and the auxiliary current. Both the main current value and the auxiliary current value can be set from 0.5 mA to 9.9 mA. The best working range of the main current is between 2 mA and 6 mA, and it's better between 1 mA and 7 mA for the auxiliary current. The system default values are 4 mA and 3 mA respectively for the main current and the auxiliary current.

Note: Don't plug HCL into HPHCL holder.

For the deuterium hollow cathode lamp (abbreviated as D_2), its lamp current can be set from 0.5 mA to 19.9 mA. The best working current is between 6 mA and 12 mA, and the system default value is 6 mA.

2) The gas control system of FAAS

The gas system of flame atomic absorption spectrometer provides enforced protective measures with a ventilation device and other security measures. Following is the diagram of the gas system (Fig. 1-1).

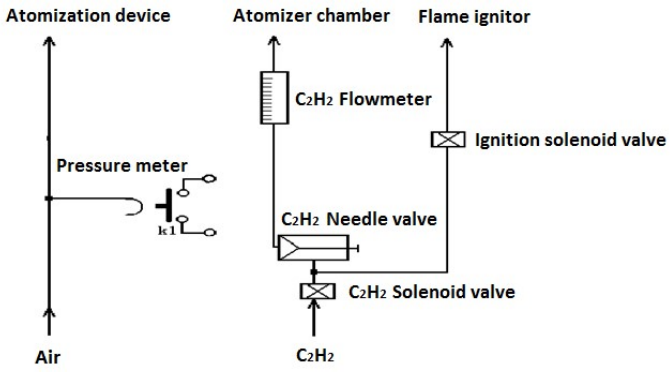


Fig. 1-1

When the electricity fails, the gas of C_2H_2 will be cut off by closing its solenoid valve, and the air will be cut off at the same time. Then, the flame in the combustion chamber will be blown out by the residual air in the pipeline, so that the instrument won't backfire, and the personal safety is ensured.

When an operational error occurs, cut off the air first, then the air pressure will decrease gradually. The air pressure switch K1 will be off when the pressure is down to about 0.06kg/cm^2, and the gas of C_2H_2 will be cut off by closing its solenoid valve, the residual C_2H_2 will burn out and the flame will be blown out by the residual air in the pipeline, and both the instrument and the personal safeties are ensured.

Nitrous oxide acetylene flame is convenient for testing high-temperature elements.The burner must be dedicated.

Following pressure settings are suggested:

Air pressure: 2.5 kg/cm^2 (0.25 MPa).

C_2H_2 pressure: 0.8 kg/cm^2 (0.08 MPa).

3) The flame atomization system

The flame atomization system includes the burner, nebulizer chamber, nebulizer and position lifting system. With the flame atomization system, the sample is atomized and ready to be analyzed.

4) The optical system

The optical system includes the first lens and the second lens, semi transparent mirror, collimating mirror, grating and slits. Most of the light from the hollow cathode lamp are focused on the grating through the optical system, then be separated to the character spectrum, and later transmit to the photomultiplier.

The reference optical path energy correction function makes the results more stable.

5) The detection system

The detection system includes the photomultiplier, preamplifier, detection amplifier, control circuit, and auxiliary circuit system. Its function is to convert optical signal to electrical signal, and the electrical signal is amplified and later be sent to the computer for further processing.

6) The mechanical control system

The mechanical control system includes the flame and graphite furnace moving platform, slit and wavelength drivers, lamp position adjustment system and burner position regulating system.

7) The graphite furnace system

The graphite furnace system consists of the graphite furnace and the power supply of heating control system. The graphite furnace includes the graphite tube, graphite cone and the electrode with water cooling system. It is designed with the shielding gas of argon inside and outside the graphite tube, which are controlled by the computer. The sample can be added into the graphite tube, and then be heated to 3000℃.

The graphite tube is the core of the atomization system for graphite furnace spectrometer, where the ground state atoms are produced. And it has a direct impact on the detection sensitivity, repeatability and other indexes. Usually, the graphite tube is made of special graphite material with high purity, high density and high strength. There are kinds of tubes such as ordinary, pyrogenic plated and plated platform graphite tubes. The detection sensitivity, repeatability and the service life of the ordinary graphite tube are worse than those of the pyrogenic plated graphite tube. The plated platform graphite tube has the best measurement repeatability. However, it is only applied to the medium and low temperature elements. The pyrogenic plated graphite tube is much suitable for medium and high temperature elements.

The detail structure of the graphite furnace body includes the graphite cone, furnace body, cooling water pipeline, shielding gas pipeline, shielded quartz window, optical sensor, temperature sensor, lock cylinder, furnace body bracket and electrodes contact.

a. Graphite cone

The graphite cone is divided into two halves, the left and the right, and its material is the same as the graphite tube. It is designed for the effective support and protection of the graphite tube. For one way, it ensures the graphite tube with ultimate reliable contact, and the contact resistance can be less than several hundreds of microampere. For another way, under its protection with inner and outer argon, the graphite tube won't be oxidized, so that its service life can be extended.

Both the left and the right parts of the graphite cone are closely fixed onto the furnace body (electrode bracket). For the dismantlement of the graphite cone, please refer to chapter 7.3.

b. Furnace body

The furnace body also contains two halves of the left and the right. And it consists of the lock cylinder and the support bracket. When it reaches the rating pressure, the graphite tube will be locked and a loop forms. The initialization conditions are prepared and the instrument is ready to carry out the procedure.

There is a water cooling pipeline in the furnace body, which is designed for rapid temperature decreasing of the graphite tube and graphite cone.

And there is a shielding gas pipeline in the furnace body, which is designed for shielding gas for graphite tube and graphite cone. The shielding gas is pure argon with the pressure of 0.3 MPa, and the flow of 1.5 L/min.

c. Shielding quartz window

The shielding quartz window is designed to avoid the argon leaking by air shielding. It also contains two halves of the left and the right. The characteristic spectrum of the element lamp passes through the shielding quartz window. The argon respectively inpours to the inner and outer at the two ends of the graphite tube. The external gas discharges from the graphite cone, and the internal gas discharges from the sampling hole of the graphite tube.

d. Optical sensor

The optical sensor is designed in the furnace body at the entrance of the light. It is used to detect the inner temperature in the graphite tube. It will send the real-time optical temperature signal to the computer when the optical sensor is chosen to monitor the temperature.

e. Temperature sensor

The temperature sensor is designed on the right furnace body. When the temperature of the furnace body reaches 80℃, the real-time message will be sent to the computer. The computer will stop the heating program automatically and alarm. The heating program also won't be carried out if the water cooling device is not switch on.

f. Lock cylinder

The graphite tube is clamped by the lock cylinder. It needs switching on or off the lock gas urn to replace the graphite tube. The lock gas urn is designed at the bottom of the furnace body. If it's not switched off or is not clamped well, the computer will alarm. Besides, the computer will record the using times of the graphite tube. User can determine to replace the graphite tube or the graphite cone according to the using times, the sensitivity decline and alarm caused by the bad contact of the graphite tube.

g. Electrode contact

The electrode contact is reliable fixed on the furnace body. It forms a loop together with above parts.

h. Furnace body bracket

Above parts are all fixed on the furnace body bracket. And the furnace body bracket is placed on the elevating gear. Adjust the elevating gear to ensure the character spectrum from the hollow lamp will all pass through the graphite tube.

8) Other auxiliary equipment (Optional)

a. Electrothermal quartz absorption tube, that is optional only for hydride generation atomic absorption spectrometry.

b. Pneumatic molecular absorption rapid injector, that is optional only for molecular absorption spectrometry.

c. Micro-volume sampler with constant current pulse, that is optional only for micro-volume sampling of atomic absorption spectrometry.

d. Auto-sampler, that is optional only for sampling of graphite furnace atomic absorption spectrometry.

2. The computer and printer

Both the computer and printer are equipped with well known brands.

3. The air compressor

The air compressor is special for the flame atomic absorption spectrometry. So far, we provide an oil free air compressor with the type of KJ-B Ⅱ that made in Tianjin.

4. The power supply of graphite furnace

The power supply of graphite furnace is special for the graphite furnace atomic absorption spectrometry.

1.3 Instrument Specifications

The instrument specifications are described in the following table:

Main AAS System	Light Source	6 Lamps automatic turret, Automatic alignment
	Optical System	Double beam Optics, fully closed optical system Large 1800 lines /mm grating reticle, 0.5nm/mm@ 200nm linear reciprocal dispersion
	Wavelength Range	180nm ~ 900nm Automatically peak find, a key optical optimization function
	Wavelength Accuracy	≤0.1nm
	Wavelength Repeatability	±0.02nm
	Spectral Bandwidth	0.1nm,0.2nm,0.4nm,1.0nm,2.0nm (5 steps with automatic changeover)
	Baseline Stability	≤±0.002A/30 min (Static) ≤±0.005A/30min (Dynamic)
	Absorbance Range	0 ~ 4 A
Flame Analytical System	Detector	Imported Photomultiplier Tube (PMT)
	Burner Head	Full titanium combustion head, 50mm or 100mm general combustion head
	Atomization Chamber	Polymer explosion-proof spray chamber
	Nebulizer	Atomizer efficient glass atomizer, can also be customized
	Flame Atomizer Positioning	Software controlled horizontal and vertical positioning
	Ignition Type	Microcomputer control, automatic ignition through software operating
	Gas Control	Automatic gas control system
	Detection Limits(Cu)	0.002μg/mL
	Precision	RSD≤0.5%
Graphite Furnace Analytical System	Heating Mode	Vertical heating
	Temperature Control Method	Vertical optical temperature monitoring graphite tube wall temperature
	Temperature Range	Room Temperature (RT) to 3000℃
	The Program	Automatic temperature control up to 20 order
	Furnace Vision System	Colored GFTV camera system
	Temperature Control	The furnace enriched up to 20 times
	Characteristics Volume	0.5×10^{-12}g (Cd)
	Detection Limit	0.4×10^{-12}g (Cd)
	Precision	RSD≤3%
	The Cooling Water	Can choose cooling water circulation system
	Safety	The graphite tube damage, water flow, air pressure and other alarm temperature overheating protection
Power Supply	AC220V (+5% ~ -10%), 60/50Hz, Lamp Current : Pulsed power supply	
Weight/Size	150 Kg / Dimensions: (L)988mm *(W)412mm* (H)440mm	
Interface	Computer and USB interface communication	
Software	PC communication software to control AAS system, Data processing, Data analysis, Print etc.	

Auto-sampler	Sample Tray	130 sample cups, 6 reagent cup
	Sample Volume	1-100µl
	The Smallest increment	0.1µl
	The Volume of Sample Repeatability Volume	better than 1% (at the time of 10mL-100mL)
	Repeated Sampling Frequency	up to 99 Times
	Cleaning Waste Container Size	each 500mL
Hydride Vapour Generator	Method	The Hydride Atomic Absorption method is used to determine trace elements such as Arsenic(As), Selenium(Se), Antimony(Sb), Bismuth(Bi), Lead(Pb), Tin(Sn), Tellurium(Te) and Germanium(Ge). The cold Atomic Absorption is used to determine Mercury (Hg).
	Pneumatic Automated Technology	Auto-sampling system, Automatic fluid measurement system, low stabilizer, Program-time controller and so on are pneumatic automatic systems which are run by the carrier gas source pressure.
	Highly Automatic	After pressing the start key, the whole process (sampling, reacting, calibrating and cleaning) will be finished automatically.
	Unique Electric Quartz Absorption Tube (Atomizer)	Compact (can be used in the Zeeman AAS), Speedy Temperature rising, easy installation, and Stable Temperature
	Sensitivity	≤1ng/ml/1%A
	Sensitivity of Arsenic(As)	≤0.15ng/ml/1%A
	RSD	≤3%
	Less Solution	Sample 2-2.5mL (including clean), Potassium borohydride 1-1.5mL, carrier liquid 5-6mL.
	Measurement Time	25 – 35 Seconds
	Power Supply	AC220V, 50/60Hz
Oil-Free Air Compressor	Air Flow	118L/min
	Speed	1400RPM
	Max. Pressure	8Bar
	Restart Pressure	5Bar
	Tank Capacity	24Litre
	Noise Level	52dB
	Power	600W
	Power Supply	AC220V, 50/60Hz
Recirculating Water Chiller	Cooling Mode	Compressor Cooling
	Temperature Controller	PID Controller
	Temperature Range	5°C – 35°C
	Accuracy	±1°C
	Cooling Capacity	1300W @25°C
	Pump Pressure	≤1.3Bar
	Pump Flow	≤15Litre/min
	Tank Volume	8 Littre
	Tank Material	Stainless Steel SS304
	Refrigerant	R134a
	Power Supply	AC220V, 50/60Hz

Chapter 2 Installation and Performance Qualification

The instrument installation should be done by our technical engineer or authorized professional agent that trained by our company. TOPLAB INDIA PVT. LTD. shall not be liable for the instrument damage or its abnormal running that caused by installation without authorizations.

Before installation, user should prepare the necessary conditions according to the requirements that described in this manual. Then, inform us to do installation and performance qualification.

2.1 Installation Requirements

1. Environment Requirements

The instrument has been designed for indoor use. A laboratory should be prepared, and it should be free of draft, dust, corrosive vapors as well as vibrations. Neither setting up the instrument near electromagnetic field, nor exposing the instrument to direct sunlight or the radiation of heaters. To obtain the best performance from your instrument, following requirements also should be met:

a) Temperature: 20℃~30℃, Relative Humidity: 10%~75%, the laboratory should equip with air conductor and dehumidification equipment.

b) Power Supply: For the main structure, a normal power supply of 220 VAC is required, and a purifying AC regulator with the total power 1kWis suggested. The power of the air compressor is about 200 W, and it's no need to connect to AC regulator, it can be connected to another 220 VAC power supply. For the power supply of graphite furnace, another 220 VAC power supply with large power of above 6 kW is required.

c) Workbench: A workbench with the dimensions of 2400 mm× 1000 mm× 800 mm (L×W×H) is suggested. The material of the workbench should be terrazzo or wood which is inflexible, shockproof, clean, and smooth. And for the convenience of maintenance, it should be placed with a clearance of at least 60 mm to the wall.

2. Ventilation System

It will produce metal vapor and toxic gases during the combustion process, which is harmful to human health. Thus, a ventilation system is required so that the harmful substance can be exhausted. And the ventilation system should be fixed above the combustion chamber. Following is a diagram of the ventilation system (Fig. 2-1):

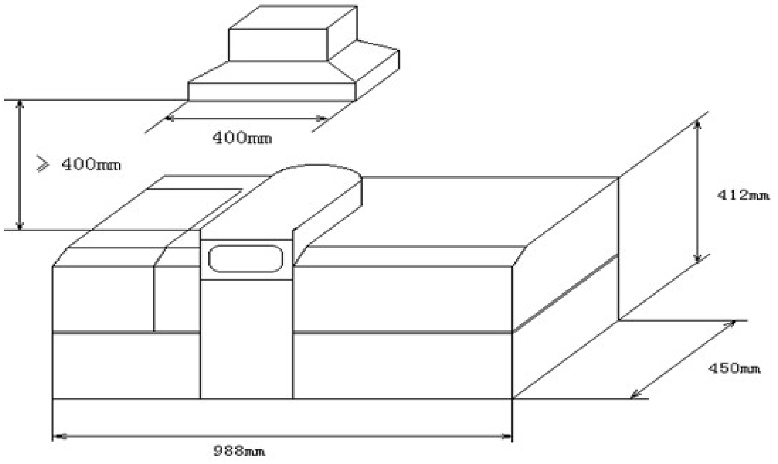


Fig. 2-1

3. Gas sources and equipments

a) Air: Air is the auxiliary gas for flame atomic spectrometry, and an air compressor is used as air equipment. The air output should be dry and clean with the pressure of 0.25 MPa.

b) C_2H_2: For flame atomic spectrometry, a purity of 99.6%~99.9% C_2H_2 gas is required, which is in white gas cylinder, and the flame should be light blue. A qualified acetylene pressure regulator should be connected to the acetylene cylinder and its output pressure should be stable at 0.08 MPa. For the acetylene cylinder, a total pressure of at least 0.3 MPa should be guaranteed. Otherwise, it should be replaced with a new one. Because there is some acetone mixed in the acetylene cylinder, when the total pressure is less than 0.5 MPa, acetone vapor may brought out with acetylene and affect the measurement accuracy and the instrument itself. The acetylene cylinder should be standing upright during the use and storage. To keep the gas balance in the cylinder and avoid the acetone brought out, don't immediately use the C_2H_2 gas when the cylinder is carried to the laboratory, and please wait at least 24 hours.

c) Argon or nitrogen: For hydride generation atomic absorption spectrometry and graphite furnace atomic absorption spectrometry, argon or nitrogen is required. And its output pressure is 0.25 MPa.

2.2 Unpacking

1. Checking

Check the outer packing after receiving the instrument immediately, if there is anything damaged, please contact the delivery company and the insurance company as soon as possible, and inform us.

If the outer packing is all right, unpack the contents carefully and check the materials according to the packing list. Please contact TOPLAB INDIA PVT. LTD. immediately if any inner part is missing.

The documents together with the instrument include the user's manual, packing list, inspection card and certificate.

2. Unpacking

A wooden packaging is used for the instrument outer packing. The instrument is packed with liner foam and with its base plate fixed on the outer packing by two angle iron and four bolts. Just remove the four bolts to unpack the instrument.

3. Packing list

Following is the packing list of the graphite furnace device:

No.	Item	Unit	Qty	Note
1	Micropipettor (50μl)	pc	1	
2	External gas tube (ϕ=6mm)	meter	5	
3	External water tube (ϕ=10mm)	meter	5	
4	Graphite cone	set	1	Specialized
5	Pyrogenic graphite tube	pc	10	Specialized
6	Elbow forceps (5 inch)	pc	1	
7	Signal cable	pc	1	Specialized
8	USB cable (1.5 meters)	pc	1	
9	Packing List of graphite furnace device	pc	1	
10	screw		4	

2.3 Installation

1. Electrical Connection

All the electrical connections of the instrument are shown as the following figure (Fig. 2-2):

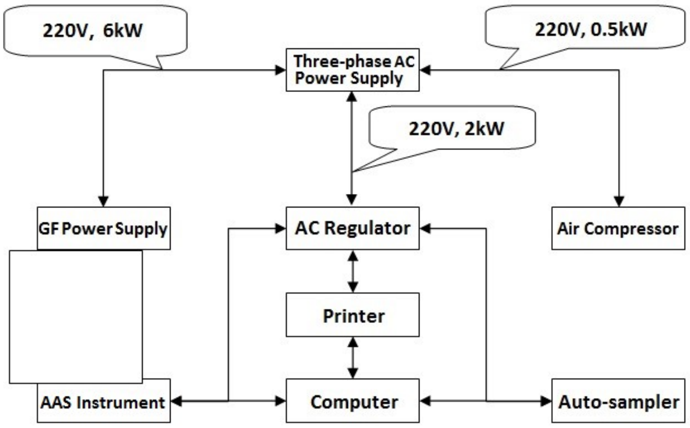


Fig. 2-2

AC Regulator: It provides stable voltage of about 220V to the instrument, computer and printer, with a fluctuation less than ±1% and a response speed between 20 ms and 100 ms.

220 V, 6 kW: It provides power to the graphite furnace.

220 V, 2 kW: It provides power to the AC regulator, which will provide stable power to the instrument, computer and printer.

220 V, 0.5 kW: It provides power to the air compressor.

Note: Be sure to connect the ground cord before the electrical connection.

The power and communication interfaces are shown as the following figure (Fig. 2-3):

Fig. 2-3

2. Gas Connection

The gas connection is shown as the following figure (Fig. 2-4):

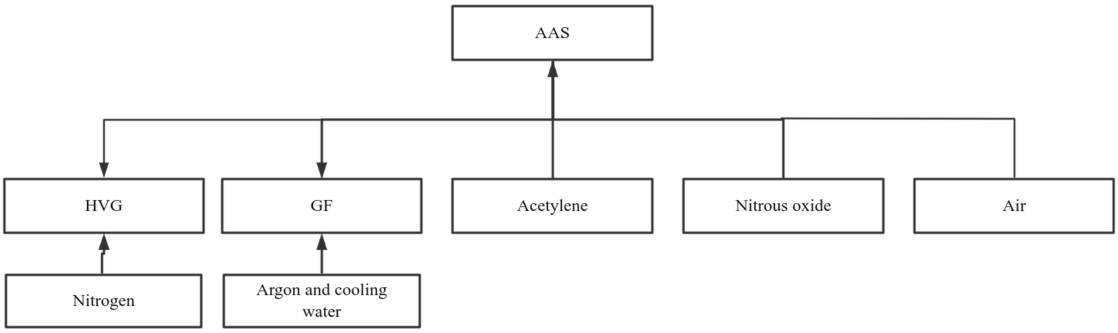


Fig. 2-4

The gas connection interfaces are shown as the following figure (Fig. 2-5):

Fig. 2-5

2.4 Performance Qualification

1. Static Performance Qualification of Flame Atomic Absorption Spectrometer

Although both the flame atomization device and the graphite furnace atomization device are at the same electric platform, the default state is that the flame atomization device is in the light path, and the graphite furnace atomization device will automatically move into the light path only when the graphite furnace atomic spectrometry is chosen.

Following are the operating steps for static performance qualification of flame atomic absorption spectrometer:

Step 1: Set the lamp position and lamp current in the condition setting interface. For normal hollow cathode lamp, the default current value is 3 mA. And for high-performance hollow cathode lamp, the default current values are 4 mA and 3 mA respectively for main current and auxiliary current.

Step 2: Set the negative high voltage for the photomultiplier.

Step 3: Set the bandwidth.

Step 4: Carry out "Lamp positioning" after completing above settings. If the set lamp position is just in the light path, then click "Cancel", otherwise, click "OK" and the lamp will turn to the new set position. The operation of "Slit positioning" is like the same.

Step 5: In the wavelength scanning interface, user can do wavelength positioning and peak searching with the characteristic wavelength of the element.

The main purpose of the static performance qualification is to obtain a stable transmittance of 100%.

The best setting of above conditions are based on the instrument's stability and sensitivity. User can refer to the conditions optimization. Here, the illustration is based on the stability. The current value of HCL should be high enough. However, it is better not to exceed 5 mA. The main current value of HPHCL is better not to exceed 5 mA, usually 3 mA~4 mA is enough. And the auxiliary current value can be set according to the actual situation, and it is better when the highest energy is obtained. The negative high voltage of the photomultiplier is better to be lower, and better to be under 250 V. The highest value is better not to exceed 300 V. The bandwidth should be wide enough to obtain better stability. It's better to select a wider bandwidth and just excluding interferences such as spectral interference. The lamp position and wavelength of the instrument are controlled by the computer. Just click "Start positioning" in the interface when above conditions are chosen, it will go to the element characteristic wavelength and search the peak value automatically. It will also go to the optimal lamp position without handle operation. Finally, click "Auto Energy" to obtain the transmittance of 100%, and then the static performance qualification based on the stability is completed.

2. External Optical Path Performance Qualification

The external optical path performance qualification (Fig. 2-6) is just to adjust the long slit of the burner, and make it below parallel to the external optical path, so that the light will pass through the atomization device at utmost and be absorbed by the sample atomic vapor during the sampling and atomization process.

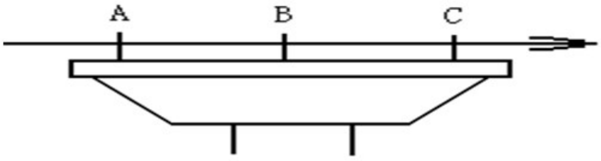


Fig. 2-6

Generally, it means the external optical path performance qualification is completed when following conditions are met.

When the transmittance of the instrument is 100%, put a small screwdriver or toothpick or other tool with a diameter between 1.2 mm and 1.5 mm respectively onto the three positions (left position A, middle position B, and right position C, as shown in above figure). First, put the tool onto the position B, and adjust the nut up and down to make the transmittance to be 0%. Then, put the tool onto the position A, and adjust the rotary rod to make the transmittance to be less than 10%. Later, put the tool onto the position C, and adjust the rotary rod to make the transmittance to be less than 30%. Otherwise, adjust the rotary rod again until it meets the requirements.

Note: Generally, there is no need to adjust the front and rear position of the burner, for it has been adjusted before delivery.

3. Dynamic Performance Qualification of Flame Atomic Absorption Spectrometer

The dynamic performance qualification is based on the static performance qualification. Then, adjust the fuel ratio and the burner height to find out the optimal condition, as to obtain much ideal determination sensitivity. The conditions are much rigorous to obtain the detection limit. Make sure that the current conditions can meet the optimization requirements.

Following are the additional operating steps for dynamic performance qualification of flame atomic absorption spectrometer:

Step 1: Nebulizer adjustment

The nebulizer of the instrument has been adjusted to a good condition, and there is no need to adjust it any more except special requirements. Following is the method of the adjustment: First, loosen the pressure valve, and remove the nebulizer from the nebulizer chamber, and spray with distilled water. The spray should be like a peacock showing its tail and mainly spray from the left side, upper side, and right side, and only a little from the below. The best situation is that the spray be fine and dense, and slightly forward. The impact ball handle within an angle of about ±45 degree below will be a best position. Then, put the nebulizer back to the nebulizer chamber. When the transmittance is 100%, spray with distilled water without flame, the transmittance declined to 5% ~ 20% is much better, and the transmittance of no more than 50% will be ok. Otherwise, user should adjust the nebulizer again or replace with a better nebulizer. The method will be inaccurate if the burner has been heated before spraying. However, user still can do measurement when the transmittance is more than 50% excluding the demand of high sensitivity.

Step 2: Ignition and sample atomization

Refer to the operation instruction, ignite the flame and the sample will be atomized. Then, adjust the fuel ratio and the burner height to find out the optimal condition, as to obtain stable signal and high detection sensitivity. If it’s still too hard to find out the optimal condition, adjust the static conditions such as lamp current, negative high voltage and bandwidth again after the flameout, then, adjust the dynamic conditions to optimal ones as required.

2.5 Conditions Optimization

1. Optimization Criteria

a) Optimization based on stability

Good detection stability is the first consideration when doing conditions optimization. And the detection sensitivity can be less considered.

b) Optimization based on sensitivity

Detection sensitivity can be the first consideration when doing conditions optimization. However, the detection stability, interferences, and other influence factors also should be considered properly.

2. Conditions Optimization

a) Lamp Current

Lamp current is one of the important conditions of static conditions optimization. For normal hollow cathode lamp, excluding some elements, the smaller the lamp current is, the higher the detection sensitivity will be. However, the smaller the lamp current is the worse the detection sensitivity will be. For high performance hollow cathode lamp, the detection sensitivity also will be improved at least dozens of percent when the detection stability is greatly improved, and sometimes even be improved for several times, which is much advantaged to improve the instrument performance. There are already twenty two kinds of HPHCL up to now, especially for hydride elements. Instead of weak and unstable HCL, all the hydride elements use HPHCL to improve the detection stability, and the detection sensitivity are improved several times. So, HPHCL is preferred as far as possible.

For HCL, the best current range is between 1mA and 5 mA, and to satisfy the stability requirement, a higher current of 6 mA ~ 7 mA is enough. For HPHCL, the best current range of main current is between

1mA and 5 mA, and the best current range of auxiliary current is between 1mA and 8 mA. Generally, the sum value of the two currents not exceeding 10 mA ~ 12 mA is much better. Another empirical data is that the auxiliary current more or less than the main current with one third would be better. It will be the optimized value when the energy reaching its peak value by adjusting (increasing or decreasing) the auxiliary current.

b) Negative high voltage

Negative high voltage is also one of the important conditions of static conditions optimization. The smaller the absolute value of the negative high voltage is, the higher the detection stability will be. The best range of the negative high voltage is between 150 V and 300 V. For much higher value, it's better not to exceed 400V.

c) Gain

Gain selection is peculiar to the static conditions optimization of this instrument. It has a special effect on those unstable and relatively weak element lamps. The negative high voltage can be lower because of the gain selection. A gain selection of two times or four times may be better for the detection stability. However, it is not absolute, and it depends on the specific element. One time gain is usually selected.

d) Bandwidth

Generally, the detection stability will be much better with a wider bandwidth, but the detection sensitivity will be worse. However, it's on the premise of that there is no spectral interference. And a wider bandwidth is preferred as far as possible.

e) Fuel Ratio

The fuel ratio is a ratio of fuel gas and auxiliary gas, and it determines the flame properties, to be a poor burning flame, or neutral flame, or a rich burning flame. Certain elements have much higher detection sensitivity with poor burning flame, but its detection stability is compromised. And some elements have much higher detection sensitivity with rich burning flame. Which element has higher detection sensitivity with certain flame, user can refer to relevant documents. Anyhow, it depends on the actual situation.

f) Burner Height

The burner height here is especially for the sensitive absorption region of the element. There are three sensitive absorption regions for atomic absorption spectrometry, 1 mm - 4 mm, 4 mm - 8 mm, and 8 mm - 16 mm. Different elements have different sensitive absorption regions. Which element has high detection sensitivity in certain absorption region, user can refer to relevant documents. The best height obtained from the experiment is the most feasible.

g) Lamp Position

The lamp position is not regarded as an optimum condition in many documents. Only in a few documents has referred, and it is adaptive to the conditions. Although its performance maybe as well as foreign products, even be much better, domestic hollow cathode lamps have larger assembling errors, and most of them are without the right luminous solid angle, so that only a few lights from the lamp can focus on the monochromator, and there is a large position deviation. Thus, it is necessary to adjust the lamp position. For the instrument has automatic lamp position adjustment function, the lamp itself can be rotated half-circle at most.

Chapter 3 Operation Instructions of FAAS

3.1 Preparations

1) Put the element lamp into the lamp holder, and remember the lamp holder No.

2) Connect the acetylene cylinder and air compressor.

3) Prepare the standard solutions and the sample solution.

3.2 Basic Operations

1. Power on

The sequence of power on is power on the computer first, then power on the instrument, while power off the instrument, the sequence is power off the instrument first, then power off the computer.

When the computer is power on and there displays a shortcut, power on the instrument, and an arrow will shown in the taskbar at the lower right corner of the desktop. Make sure the arrow is already existed. Otherwise, the software can’t be opened.

2. Power off

When the measurements are completed, and user wants to power off the instrument, click the arrow at the lower right corner first, and a prompt of “Stop USB device CH372/CH375” will be shown. Click the arrow again and a prompt of “Safely remove the hardware” will be shown. Then, the instrument is ready for power off.

3. Enter the software interface

Double click the shortcut icon on the desktop and enter the software initial interface (Fig. 3-1 ?). Click any key to enter the main interface (Fig. 3-2).

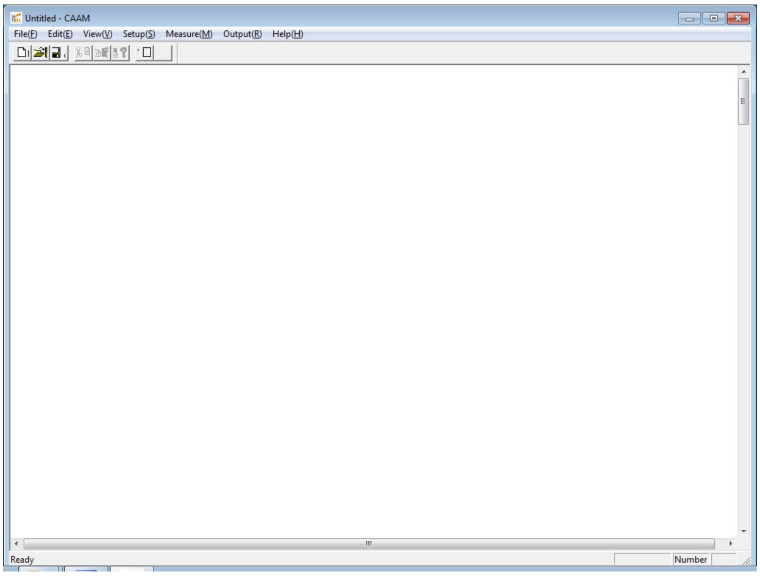
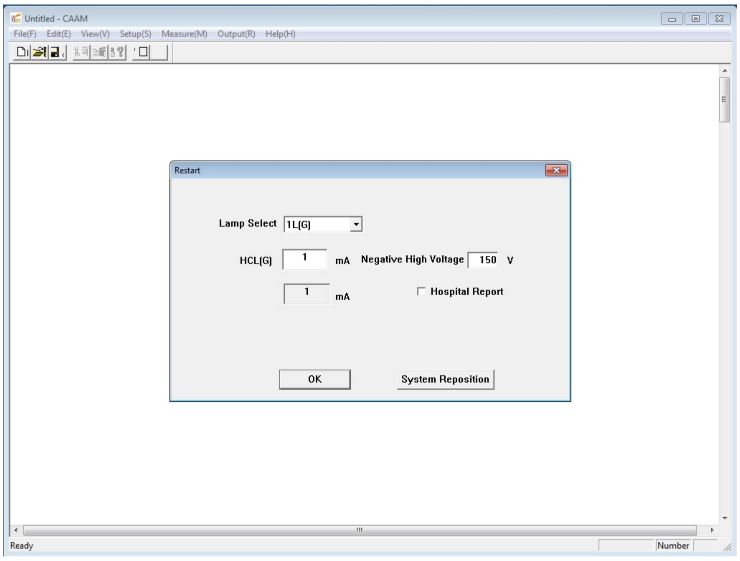


Fig. 3-2

4. System reset

The instrument setting includes system reset, stability, optical debugging, and electrical debugging. And the interface of system reset is shown as in following figure (Fig. 3-3).



Lamp Select 1L(G)

HCL(G) 1 mA Negative High Voltage 150 V

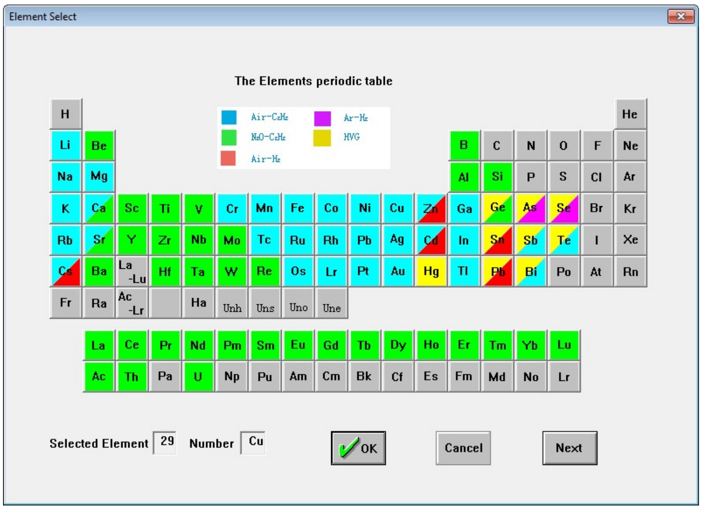
1 mA ☐ Hospital Report

Fig. 3-3

Select the lamp position (lamp holder number) first, then check the lamp position to make sure it is in the light path. Otherwise, rotate the lamp and make it to be in the light path, and click "System Reset". Then, click "OK", all settings will revert back to defaults. Generally, for every day measurement with the same element, there is no need to do system reset each time. One time reset is precise enough for measuring several elements in the same day. Measurement in several days without system reset is also ok. However, the system reset should be done when the wavelength deviation is more than 0.2 nm.

5. Element selection

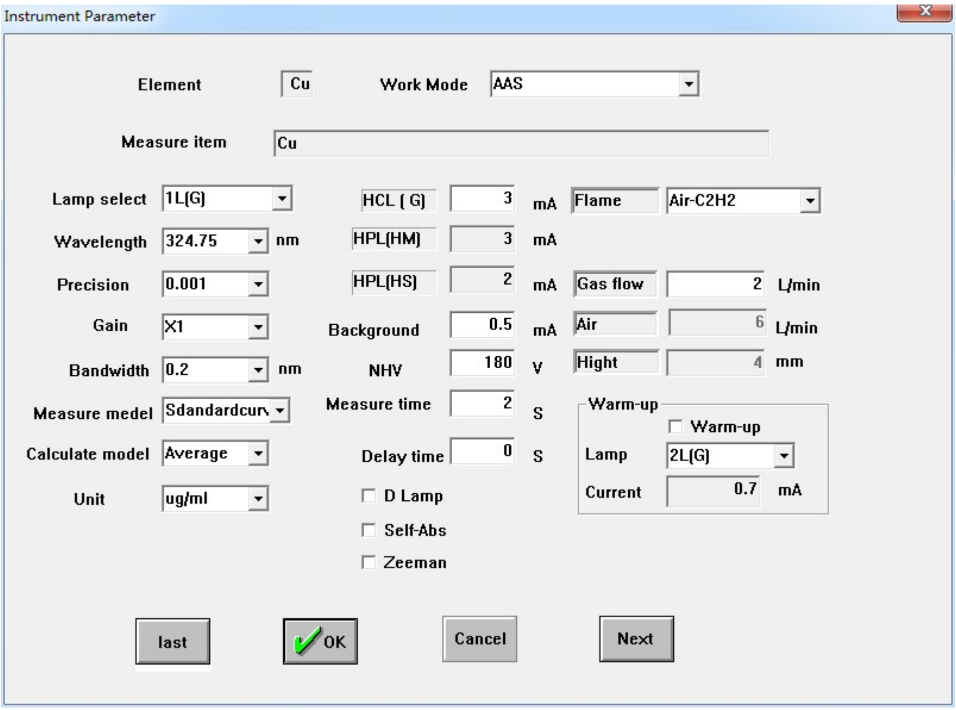
Click "Sample selection" in the menu of "Measurement" in the main software interface (Fig. 3-2), and a periodic table of elements will be shown (Fig. 3-4).



Selected Element 29 Number Cu

Fig. 3-4

Select the element to be measured. The chemical symbol and atomic number of the element will be shown below the periodic table of elements. Click “OK” to back to the main interface. User also can click “Next” to enter the parameters setting interface (Fig. 3-5).



Element Cu
Work Mode AAS
Measure item Cu
Lamp select 1L(G)
Wavelength 324.75 nm
Precision 0.001
Gain X1
Bandwidth 0.2 nm
Measure medel Sdandardcurv
Calculate model Average
Unit ug/ml
HCL (G) 3 mA
HPL(HM) 3 mA
HPL(HS) 2 mA
Background 0.5 mA
NHV 180 V
Measure time 2 S
Delay time 0 S
☐ D Lamp
☐ Self-Abs
☐ Zeeman
Flame Air-C2H2
Gas flow 2 L/min
Air 6 L/min
Hight 4 mm
Warm-up
☐ Warm-up
Lamp 2L(G)
Current 0.7 mA

Fig. 3-5

6. Parameters setting

There are four operation modes including flame atomic absorption spectrometry, hydride generation atomic absorption spectrometry, atomic emission spectrometry, and graphite furnace atomic absorption spectrometry. It will show in white background when one operation mode is chosen. And other parameters such as fuel flow, burner height also will show in white background and are available for rewrite. Parameters shown in white background together with the drop-down arrow are available for option or rewrite. Then, click “OK” or “Next”, and the settings will take effect.

For flame atomic absorption spectrometry, the operation of parameters setting is as following: First, select flame atomic absorption spectrometry as the operation mode in the parameters setting interface. Then, set relevant parameters.

1) Lamp position selection

There are six lamp holders on the light rack for position selection. Lamp holders from No. 1 to No. 4 are for normal hollow cathode lamps (abbreviated as HCL), and lamp holders of No.5 and No. 6 are for high-performance hollow cathode lamps (abbreviated as HPHCL). The lamp will be lit up only when the lamp position is selected and it will transfer to the light path automatically. Please do not plug HCL into HPHCL holder, otherwise, once the position is selected, it will cause damage to the circuit board.

2) Wavelength setting

Click the drop-down arrow in the wavelength column, and select the most sensitive wavelength or secondary sensitive wavelength (if it has) of the element.

3) Display precision setting

Click the drop-down arrow in the precision column, and there are two selections, 0.001 and 0.0001. Generally, precision of 0.001 is as the default. For high measurement requirements, or measure with trace elements, user can select the high precision of 0.0001.

4) Gain setting

Click the drop-down arrow in the gain column, and there are three selections, ×1, ×2, and ×4. Generally, the default gain is ×1. The gains of ×2 and ×4 are designed for negative high voltage reduction, and for detection stability improvement or electronic noise reduction. Anyhow, select a good static electronic condition to reduce the total noise and improve the detection limit.

5) Bandwidth selection

Click the drop-down arrow in the bandwidth column, and there are four kinds of bandwidth for chosen, 0，0.2 nm, 0.4 nm, 1.0 nm and 2.0 nm. Generally, the default bandwidth is 0.2 nm. The selections of 0.4 nm, 1.0 nm and 2.0 nm are usually for improving the light intensity and the detection stability. A wider bandwidth is necessary especially for those elements neither with adjacent spectral interference nor with strong intensity itself. User can select a wider bandwidth when a high performance hollow cathode lamp is used. “Close the bandwidth” is only for factory adjustment.

6) Flame style selection

Click the drop-down arrow in the flame style column to select the flame style. Acetylene air and acetylene nitrous oxide are available.

7) Measurement method selection

Click the drop-down arrow in the measurement method column, and there are three methods for chosen, standard curve, standard addition, and absorption photometry. The former two methods are commonly used for concentration measurement, and the standard curve is used more commonly, and usually be a default method. However, the method of absorption photometry can be selected when there is no mature method and it needs method experiments, and user can directly enter the sample measurement interface to modify kinds of conditions, that including electronic conditions, fuel ratio, burner height, and so on, so that to obtain the optimized conditions for certain element in the particular environment. Then, measure the sample with one of the former two methods and obtain the best result.

8) Calculation method selection

Click the drop-down arrow in the calculation method column, and there are three calculation methods for chosen, average, peak height, and peak area. The flame atomic absorption spectrometry and the atomic emission spectrometry are commonly use average as the calculation method. In some special cases, for example, when using the pulse sampling device, the calculation method of peak height, or peak area should be used, while adopting the hydride generation atomic absorption spectrometry or the graphite furnace atomic absorption spectrometry, the calculation method of peak height, or peak area should also be used. If none of above calculation methods is selected, it will calculate with average. And for hydride generation atomic absorption spectrometry and graphite furnace atomic absorption spectrometry, peak height is used as a default calculation method.

9) Concentration unit setting

Click the drop-down arrow in the concentration unit column, and there are six units for chosen, µg/ml, mg/ml, µg/L, mmol/L, µmol/L, and %. And µg/ml is commonly used as the default concentration unit.

10) Lamp current setting

The default lamp currents are 3 mA, 4 mA, 3 mA and 6 mA respectively for HCL, the main current of HPHCL, the auxiliary current of HPHCL, and D_z. If the lamp positions of No. 5 and No. 6 are not selected, both the setting columns of the main current and the auxiliary current for HPHCL will be grey, and they won’t take effect until the lamp position is selected. Above three types of lamps are controlled by the computer and their currents are given through D/A just with the initial setting values, and they need to be further adjusted in the wavelength scanning interface.

11) Negative high voltage setting

The initial value of the negative high voltage is also give through D/A. It rests with the lamp position of the element and its emission energy. If the energy is weak, the absolute value of the negative high voltage should be large. Otherwise, it should be small. The initial value is usually between 150 V and 300 V, and only a few elements with the value of above 300 V. For some element, if the negative high voltage is too high, it can be adjusted by widening the bandwidth or enlarging the gain, to reduce the negative high voltage, then to obtain stability and little instrument noise.

12) Measurement time setting

For flame atomic absorption spectrometry, molecular absorption spectrometry, and atomic emission spectrometry, the measuring time of 2 s is enough, and 2 s is usually as the default. For specific requirement, it can be chosen in between 0.2 s and 20 s.

13) Delay time setting

The range of the delay time is between 0 s and 20 s, and it is commonly used with hydride generation atomic absorption spectrometry, graphite furnace atomic absorption spectrometry and atomic absorption spectrometry with pulse sampling. Generally, 0 s is as the default.

14) Fuel gas, carrier gas and burner height settings

The three conditions of fuel gas, carrier gas and burner height are adopting the optimal values obtained from each adjustment interface. Then, they will be saved in the memory of the computer. And it's convenient for reference in the next use with the same method

15) Measurement repetition setting

The measurement repetition can be selected between 1 times and 30 times, and 1 time is commonly used as the default.

16) Standard sample number setting

Generally, three points is enough for establishing a standard curve. User can select the standard sample number in between one point and 8 points that excluding zero. Three points is commonly used as the default.

17) Background correction method selection

The Atomic Absorption Spectrometer can be used with or without background correction. There are 3 background correction methods for chosen, D_2 lamp background elimination, and self-absorption background deduction and Zeeman background correction. AAS with D_2 lamp background elimination is commonly used. If the background correction method with D2, Self or Zeeman background elimination is selected, the measurement will be done with this background correction method.

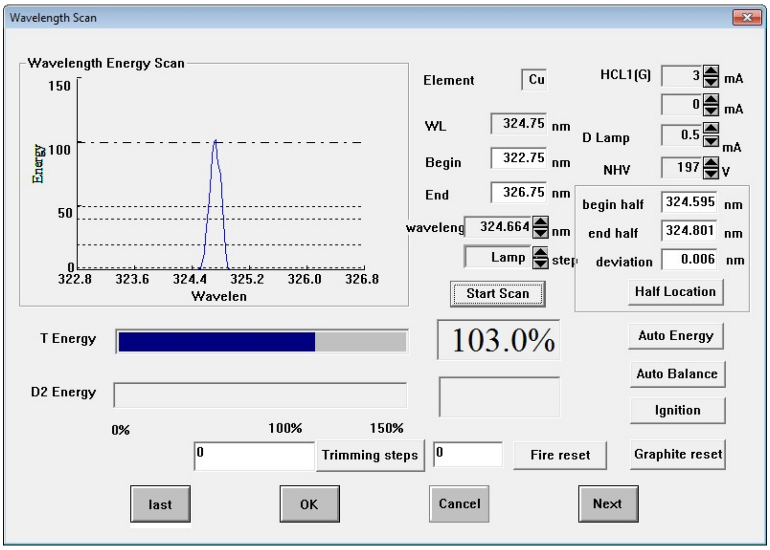
18) Other settings

When above settings are completed, user can click "lamp positioning", if the lamp position is already in the light path, just click "Cancel" to cancel the operation (Fig. 3-6).

Do you want to circumgyrate lamp?

Fig. 3-6

The operation of “Slit positioning” is like the same. If user wants to change the slit, click “OK”, otherwise, click “Cancel”. If pre-heating is selected, just input the lamp holder number that to be pre-heating. However, the pre-heating current is limited. After completing all the settings, user can click “Next” to enter the wavelength scanning interface (Fig. 3-7).



Element Cu
WL 324.75 nm
Begin 322.75 nm
End 326.75 nm
waveleng 324.664 nm
HCL1(G) 3 mA
0 mA
D Lamp 0.5 mA
NHV 197 V
begin half 324.595 nm
end half 324.801 nm
deviation 0.006 nm
103.0%
0 Trimming steps
0

Fig. 3-7

7. Wavelength scanning

User can exactly find out the wavelength peak by wavelength scanning, and following are the operation steps.

Step 1: Set the start wavelength and the end wavelength. Generally, the default range is set with ± 2 nm around the most sensitive wavelength. User can set the values as remanded respectively after clicking in the blank of the start wavelength and the end wavelength.

Step 2: Set appropriate negative high voltage after setting the lamp current. Generally, a range of 150 V - 300 V can meet the requirement. Certain elements may require a higher range of 300 V - 400 V, which is not suggested. Here, the negative high voltage can be adjusted together with the gain and the bandwidth.

Step 3: Start wavelength scanning. User can click “Start Scanning” to start the wavelength scanning when the element lamp is already in the light path. If the settings of lamp current and negative high voltage are appropriate, and the lamp is right in the light path, and the scanning energy is just

among 60% ~ 110%, the wavelength motor will return to the peak position after completing the scanning in the setting range, without displaying the peak value. Then, the lamp position motor will automatically start scanning step by step near the lamp position, finally stop at the optimal position and the transmittance value will be given. Generally, it will give the peak value after wavelength scanning and automatic fine tuning of the lamp position, and there is no need to adjust again.

However, there may be a deviation of one or two steps with the wavelength tuning, especially after a long time using. The transmittance of the wavelength peak may not be 100%, then, “Auto Energy” should be clicked, so that the negative high voltage will be automatically adjusted to reach the 100% transmittance.

If the lamp current and the negative high voltage are too high or too low, the transmittance will far exceed 100% or below 50% after wavelength positioning, the system will automatically reduce or increase the negative high voltage, and do wavelength scanning again. It may adjust for several times until it is in the appropriate position and the transmittance is between 110% ~ 60%.

Note: Following is an extreme incase that should be avoided. The element lamp is not right in the light path and its characteristic spectrum can’t focus on the monochromator, thus, the zero order light and the target wavelength can’t be found. If there is no wave form among several times scanning, and the negative high voltage rises to a much high value, please click “Cancel” immediately and return to the parameters setting interface. Reset the negative high voltage with a more appropriate value, rotate the element lamp again and make the light to be in the light path. Then, do system reset. Repeat the wavelength scanning to find out the peak after the zero order light is found.

Another case also should be avoided. When a wrong scanning range is given, the most sensitive wavelength will be not in the range, then, click “Cancel” immediately to stop the scanning, reset the scanning range, and do wavelength scanning again to find out the wanted peak wavelength.

Note: Neither misplacing the element lamp, nor with error lamp position, it should be avoided.

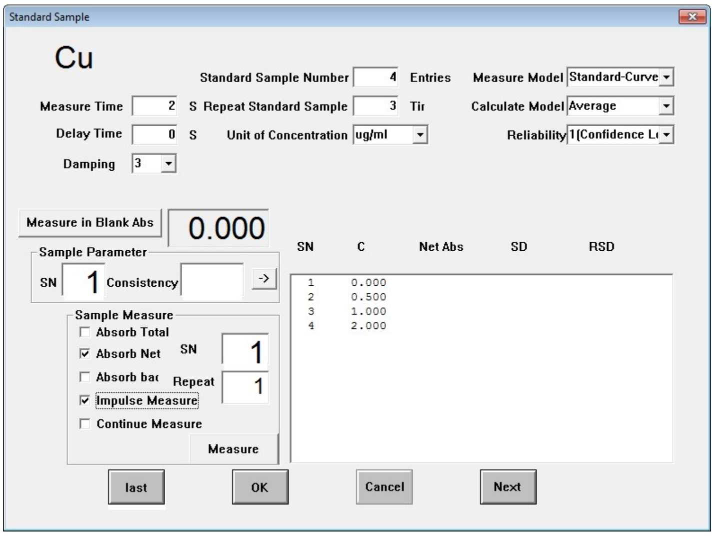
The operation of finding the most sensitive wavelength is not only applicable to atomic absorption spectrometry, but also applicable to other methods.

8. File creation

Click “OK” to return to the main interface after finding the peak wavelength, then, click “Save” to enter a file creation window, input the file name and save the file to certain file path.

9. Standard curve establishing

Select “Standard sample” in the menu of “Measurement” in the main software interface and enter the standard sample setting interface (Fig. 3-8).



Cu

Standard Sample Number 4 Entries
Measure Model Standard-Curve
Measure Time 2 S
Repeat Standard Sample 3 Tir
Calculate Model Average
Delay Time 0 S
Unit of Concentration ug/ml
Reliability 1(Confidence L
Damping 3

0.000

Sample Parameter
SN 1 Consistency

Sample Measure
☐ Absorb Total
☑ Absorb Net
☐ Absorb bac
☑ Impulse Measure
☐ Continue Measure
SN 1
Repeat 1

SN	C	Net Abs	SD	RSD
1	0.000			
2	0.500			
3	1.000			
4	2.000			

Fig. 3-8

1) Element displaying

The element is already selected in the elements periodic table and can’t be modified in the standard sample setting interface. Here it just shows which element is to be measuring.

2) Standard sample number setting

User can set the standard sample number according to the actual needs. The largest number is ten, and three is commonly used as default.

3) Measurement method setting

There are three methods for chosen, standard curve, standard addition, and absorption photometry. Generally, the default method is standard curve.

4) Measurement time setting

User can set the wanted measurement time. For flame atomic absorption spectrometry, the measuring time of 2 Sec is usually as the default.

5) Measurement repetition setting

The standard sample measurement repetition can be set between 1 times and 30 times, and 1 time is commonly used as the default.

6) Calculation method setting

There are three calculation methods, which are already described ahead.

7) Ignition

After completing the standard sample setting, return to the wavelength scanning interface to do ignition operation. For the ignition will affect the transmittance, especially for the short wavelength element, the influence is much great, and even with a 60% transmittance reduction. So that it should be complemented to 100% by energy auto-adaptation.

Before ignition, user should turn on the air compressor and the needle valve first. Then, turn on the acetylene cylinder, and it means the gas system is ready for ignition when the indicator lamp is on. Turn on the acetylene needle valve with the left hand and make the acetylene flow to reach1.2 L/min, and with the right hand pressing the left mouse button and make the cursor moving to “Ignition”, the auto-igniter will ignite the torch and burner. Then, immediately release the left mouth button when the ignition is successful. When the sample measurement is completed, turn off the acetylene cylinder, or turn off the acetylene needle valve by right rotation, the flame will go out.

Note: Some air may still exist in the acetylene pipeline and the flame can’t be ignited during the first ignition. User should continuously click “Ignition” until the flame is ignited. If the torch doesn’t work, please turn off the acetylene needle valve immediately so to avoid the acetylene leak. Check the ignitor and its controlling electromagnetism valve, and make sure that it can smoothly ignite the fire and the torch works well, then, turn on the acetylene needle valve till the flame is ignited.

8) Nebulizer cleaning

Generally, sampling with distilled water and burn for 5 min ~15 min after completing the sample measurement, so that to reduce the sample residue and the corrosion of the burner and the nebulizer chamber.

9) Concentration unit setting

There are six units for chosen, and μg/ml is commonly used as the default concentration unit.

10) File name

The file name can't be modified in this interface.

11) Standard sample concentration setting

The serial number and the concentration value of the standard sample should be corresponding. And they should be input one by one.

12) Standard sample measurement parameters setting

a) Total absorbance: It's equal to the net absorbance when there is no background absorption. Usually, it is not selected in the case of without background.

b) Net absorbance: It's a pure absorbance with the total absorbance subtracting the background absorbance, and it is always selected.

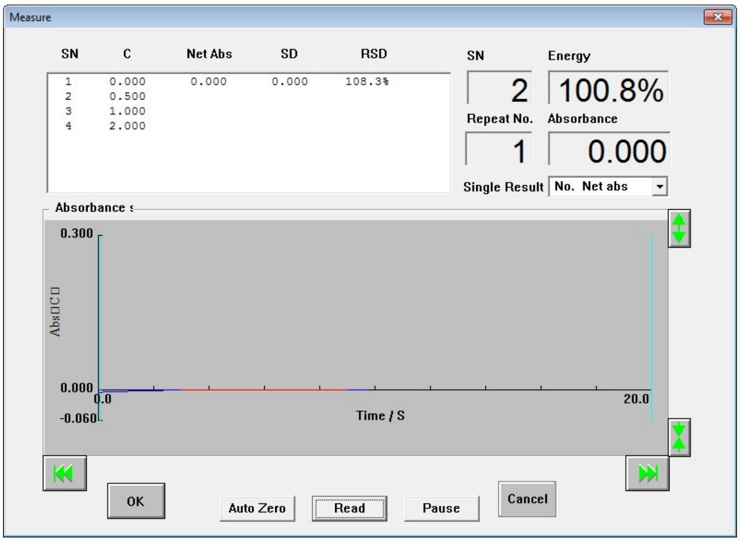
c) Background absorbance: It is commonly selected when the background absorption exists.

13) Blank measurement

Whether to do blank measurement or not, it rests on the demand. If the blank measurement is done, the sample measurement result should subtract the blank value.

14) Standard sample measurement

Click "Measure" to enter the measurement interface (Fig. 3-9).



Measure

SN	C	Net Abs	SD	RSD
1	0.000	0.000	0.000	108.3%
2	0.500			
3	1.000			
4	2.000			

SN: 2

Energy: 100.8%

Repeat No.: 1

Absorbance: 0.000

Single Result: No. Net abs

Fig. 3-9

a) Click “Auto Zero” before standard sample measurement.

b) Adapt Y-axis according to the measurement value.

c) Measure the standard samples according to the prompts.

d) Click “OK” and switch to the standard sample setting interface. Then, user can view the measurement data of each standard sample.

e) Click “Next” to switch to the standard curve interface. Then, click “OK” and a standard curve will be shown (Fig. 3-10).

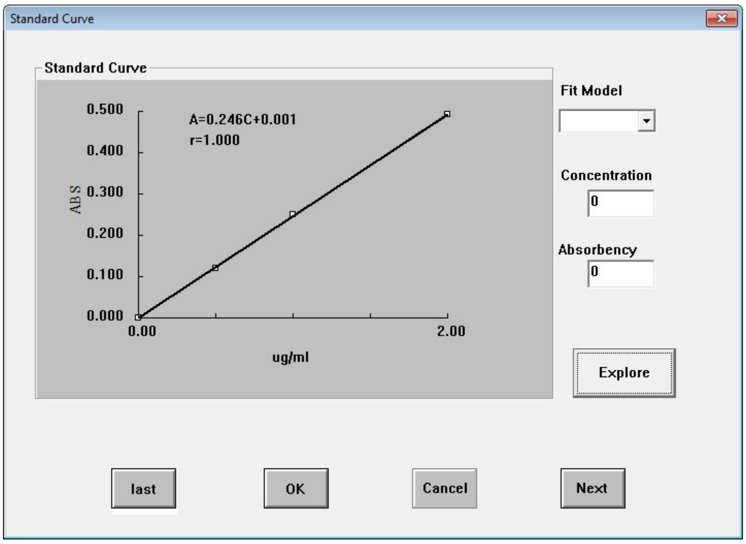


Fig. 3-10

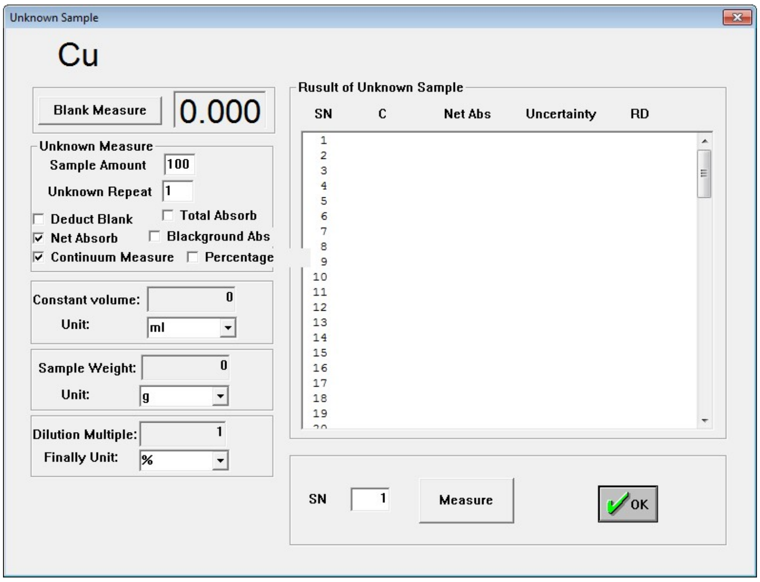
15) Fitting method

There are four fitting methods for chosen, linear fit, quadratic fit, cubic fit and quartic fit. The linear fit is usually for those curves with good linearity, while quadratic fit is chosen for those curves with a little worse linearity. If the linearity is much worse, cubic fit or quartic fit can be chosen.

User can check the relationship of each standard point with the cursor pointing to the concentration axis. Intuitively say, the value of the relationship between 0.999 and 1 is the best, and usually, it is available for sample measurement when it reaches 0.995. However, it depends on the demand, different elements, different concentration units and different conditions may have different requirements, and it can’t be all included.

10. Sample parameters setting

Click “OK” in the standard curve interface and return to the main interface. Then select “Sample” in the menu of “Measurement” and enter the sample setting interface (Fig. 3-11).



0.000

Unknown Measure
Sample Amount 100
Unknown Repeat 1
☐ Deduct Blank ☐ Total Absorb
☑ Net Absorb ☐ Blackground Abs
☑ Continuum Measure ☐ Percentage

Constant volume: 0
Unit: ml

Sample Weight: 0
Unit: g

Dilution Multiple: 1
Finally Unit: %

Rusult of Unknown Sample

SN	C	Net Abs	Uncertainty	RD
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				

SN 1

Fig. 3-11

1) Sample amount setting

Up to 999 samples can be set, and user can set the sample amount again after completing 999 samples measurement. To avoid unnecessary blank rows in the final report, better to set the sample amount as demand in the current measurement.

2) Measurement repetition setting

The measurement repetition can be set at will, and one time is used as default.

3) Blank deduction

The default situation is that the option be selected, and the blank will be deducted.

4) Total absorbance

This option is not selected as a default. If it is selected, the total absorbance will be calculated. However, it’s equal to the net absorbance when there is no background absorption.

5) Net absorbance

This option is selected as a default, and the net absorbance will be calculated and shown in the datasheet.

6) Background absorbance

The background absorbance will be calculated when it is selected. Commonly, it is not selected as a default.

7) Serial number

“SN” here is for serial number of the sample. If the sample needs to be measured again, or the sequence of the measurement needs to be altered, user can modify the serial number.

8) Continuous measurement

The sample can be measured continuously or discontinuously, and discontinuous measurement is used as default.

9) Percentage content

When "percentage" is selected, user should fill in the constant volume, the sample weight and dilution multiple. And proper unit should be selected.

11. Sample measurement

Click "Measure" in the sample setting interface (Fig. 3-11) & return to the measurement interface (Fig. 3-9).

1) Click "Auto Zero" before sample measurement.

2) Measure the sample according to the serial number and repetition.

3) Click "OK", it will switch to the sample setting interface, and the measurement result will be shown in the datasheet. User can view the sample data.

12. Operations for standard addition method

The standard addition method is another commonly used method for concentration measurement that based on the standard curve. When the sample matrix affects the measurement and the influence is not easy to eliminate, or the standard solution is difficult to prepare, or the sample amount is small, the standard addition method will be a better selection.

Select "Standard addition" in the parameters setting interface, and following are the operation steps.

Step 1: Respectively add the sample solutions with the same amount to three or four volumetric flasks.

Step 2: Excluding one of above volumetric flasks, respectively add the standard solutions sequentially with different proportions to other volumetric flasks. Prepare the blank solution at the same time.

Step 3: Respectively dilute to the constant volume with blank solution, so that a group of sample solutions with different portion of standard solution is prepared as following: Blank, sample, sample + 1 part of standard sample, sample + 2 parts of standard sample, sample + 3 parts of standard sample.

Step 4: Input the exact concentration values of above standard samples.

Step 5: Measure the blank solution and the group of sample solutions, and a standard curve will be established.

Step 6: For the standard curve doesn't pass through the origin, extrapolate the standard curve according to the absorbance of the blank solution, and the concentration value obtained from the intersection with the abscissa is just the sample concentration (Fig. 3-12).

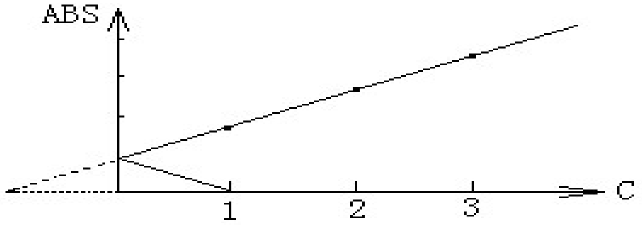


Fig. 3-12

Step 7: Review the measurement result.

Note:

a) All the measurements should be with a good linearity.

b) The standard solution for addition could not be too thick, and with the concentration between one times and four times more than the sample's is much better.

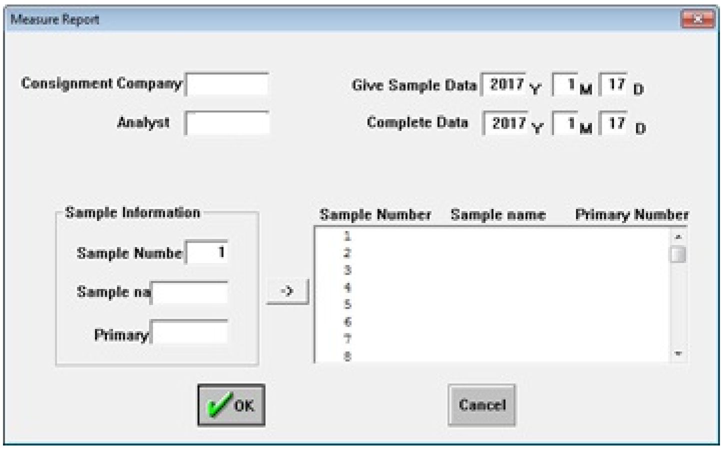
c) Compared with the standard curve method, the slope error of the standard addition method should be less than 20%. Otherwise, it means serious interference existing and the analysis accuracy will be reduced. And a further analysis is needed after eliminating the coexisting substance.

d) The sample without adding the standard solution should be measured in each group of measurements.

13. Output

1) Measurement report

Click "Output" in the menu bar of the main interface, and select "Measurement Report", a measurement report will be shown (Fig. 3-13). Fill in the information such as the sample number, sample name, client, analyst, sample deliver date, and sample measurement date, then click "OK", a further report will be shown (Fig. 3-14).

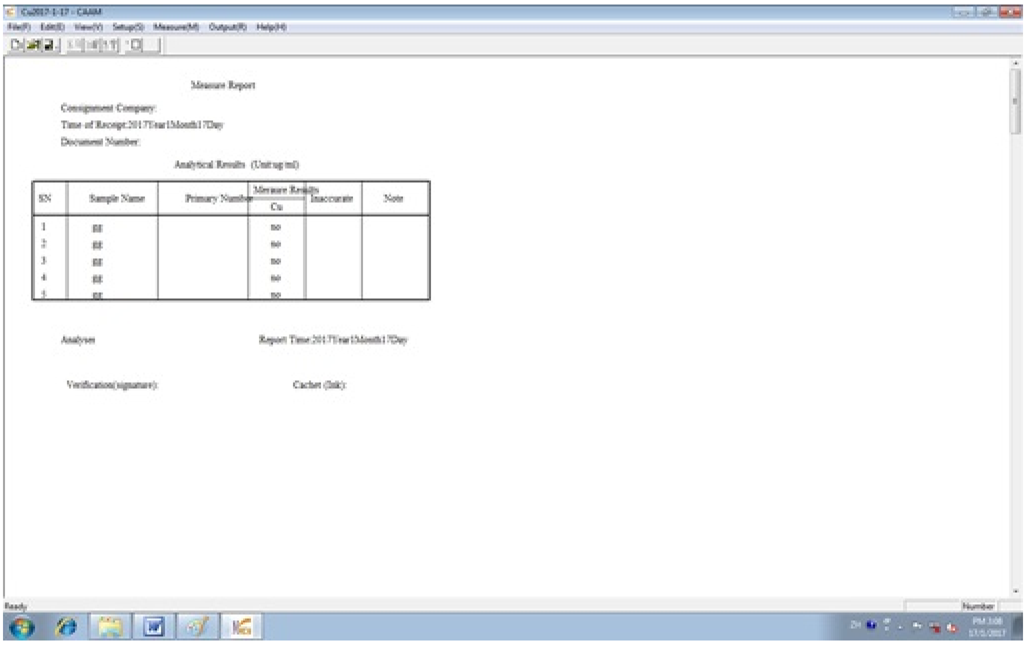


Give Sample Data 2017 Y 1 M 17 D

Complete Data 2017 Y 1 M 17 D

Sample Numbe 1

Fig. 3-13



Measure Report

Consignment Company:
Time of Receipt:2017Year1Month17Day
Document Number:

Analytical Results (Unit:ug/ml)

SN	Sample Name	Primary Number	Measure Results	Inaccurate	Note
			Cu		
1	ee		80		
2	ee		80		
3	ee		80		
4	ee		80		
5	ee		80		

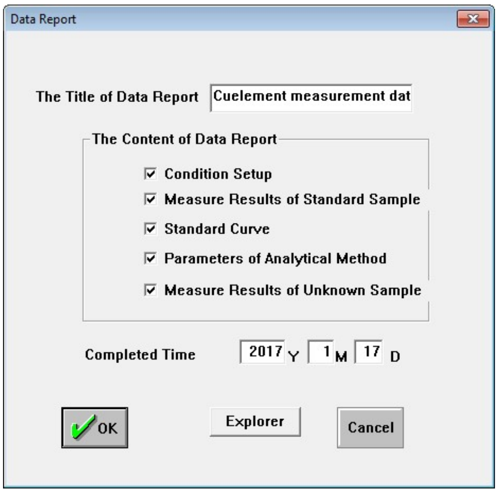
Analyser:
Report Time:2017Year1Month17Day

Verification(signature):
Cachet (Ink):

Fig. 3-14

2) Data report

Select “Data Report” in the menu of “Output” in the main software interface, a data report will be shown (Fig. 3-15).



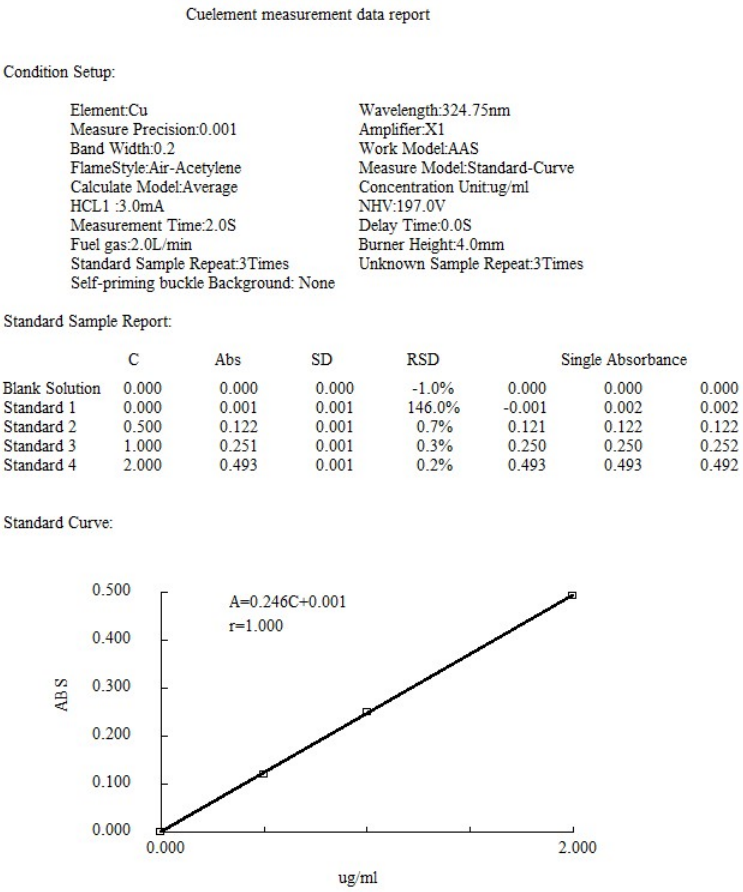
The Title of Data Report: Cuelement measurement dat

The Content of Data Report
☑ Condition Setup
☑ Measure Results of Standard Sample
☑ Standard Curve
☑ Parameters of Analytical Method
☑ Measure Results of Unknown Sample

Completed Time: 2017 Y 1 M 17 D

Fig. 3-15

There are five items for chosen as the content of the data report. And the selected content will be shown in the data report. Following is a detail data report (Fig. 3-16), which is saved by the operator and can be used for reference, and for similar test, this method can be used and it will provide great convenience to the operator.



Cuelement measurement data report

Condition Setup:

Element:Cu
Measure Precision:0.001
Band Width:0.2
FlameStyle:Air-Acetylene
Calculate Model:Average
HCL1 :3.0mA
Measurement Time:2.0S
Fuel gas:2.0L/min
Standard Sample Repeat:3Times
Self-priming buckle Background: None

Wavelength:324.75nm
Amplifier:X1
Work Model:AAS
Measure Model:Standard-Curve
Concentration Unit:ug/ml
NHV:197.0V
Delay Time:0.0S
Burner Height:4.0mm
Unknown Sample Repeat:3Times

Standard Sample Report:

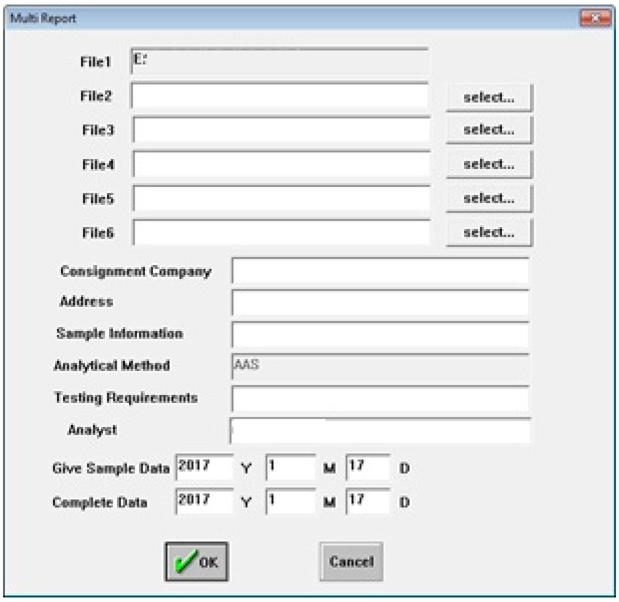
	C	Abs	SD	RSD	Single Absorbance		
Blank Solution	0.000	0.000	0.000	-1.0%	0.000	0.000	0.000
Standard 1	0.000	0.001	0.001	146.0%	-0.001	0.002	0.002
Standard 2	0.500	0.122	0.001	0.7%	0.121	0.122	0.122
Standard 3	1.000	0.251	0.001	0.3%	0.250	0.250	0.252
Standard 4	2.000	0.493	0.001	0.2%	0.493	0.493	0.492

Standard Curve:

Fig. 3-16

3) Multi report

If two or more elements need to be analyzed for the same client, a multi report will be used. Following is an example of the multi report (Fig. 3-17).



Multi Report

File1 E:
File2
File3
File4
File5
File6
Consignment Company
Address
Sample Information
Analytical Method AAS
Testing Requirements
Analyst
Give Sample Data 2017 Y 1 M 17 D
Complete Data 2017 Y 1 M 17 D

Fig. 3-17

Chapter 4 Operation Instructions of HG-AAS

For hydride generation Atomic Absorption Spectrometry, a Hydride Vapour Generation device should be equipped with the main AAS instrument.

When hydride generation atomic absorption spectrometry is selected in the parameters setting interface, the interface will be a little different, the items such as fuel gas flow, flame type, molecular absorption will turn grey and become invalid, while the carrier gas flow becoming valid.

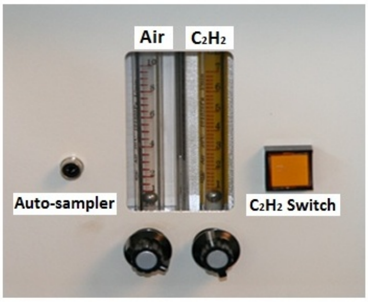


Fig. 4-1

Most of the operations are like the same with flame atomic absorption spectrometry, just plug the outlet line of the hydride generation device into the auto-sampler jack on the panel of gas path (Fig. 4-1), and connect the hydride generation device to the instrument. Following are the different operations:

1. Atomization mode

The atomization of hydride generation atomic absorption spectrometry with pneumatic component is carried out by electrothermal quartz absorption tube. Heat the electrothermal quartz absorption tube with 110V DC, and the temperature is about 900 ℃. The sample solution mixes with potassium borohydride solution and a reaction will take place. The generated gas mixture will be carried into the electrothermal quartz absorption tube and be atomized. Then, the atomized vapor will be absorbed by the characteristic spectrum of certain element.

Following are the operations for the external optical path performance qualification of hydride generation atomic absorption spectrometer:

Adjust the instrument to obtain a transmittance of 100% first. Then, place the electrothermal quartz absorption tube onto the burner, and following is the installation diagram of the electrothermal quartz absorption tube (Fig. 4-2). Plug its power cord into the power socket. Adjust the burner position with up and down, back and forwards, left and right, and make the light spot focus on the electrothermal quartz absorption tube, and then the light pass through the center. Adjust the transmittance to be 100% again.

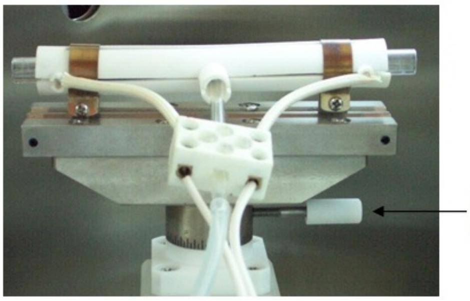


Fig. 4-2

2. Sampling method

The sample together with KBH_4 is quantitatively and steadily carried into the reaction tube by the carrier gas and carrier solution, and the generation is carried into the gas-liquid separation tube, then the gas mixture is carried into the electrothermal quartz absorption tube and atomized, and the waste liquid is drain out. For the absorption is instantaneous, the instrument reading commonly uses peak height.

Following are the preparations for sampling:

1) Put the capillary tube of the carrier solution into the carrier solution container.

2) Put the sample capillary tube into the sample container.

3) Put the capillary tube of the KBH_4 solution into the KBH_4 container.

4) Put the waste liquid tube into the waste liquid container without water sealing.

5) Connect the electric source of the electrothermal quartz tube to the outlet of the power.

6) Connect the gas tube at the rear of the hydride generation device to the pressure reduction valve of the argon cylinder.

For detail operation instructions, please refer to the operation manual of the hydride generation device.

3. Atomization optimization

The atomic absorption area of the hydride generation element uses electrothermal quartz absorption tube, whichever the hydride element is, the generated gas mixture will be atomized and absorbed in the electrothermal quartz absorption tube.

For analysis with hydride generation atomic absorption spectrometry, to obtain the optimal absorption, the whole transmitted energy should completely pass through the electrothermal quartz tube, and it can be done by adjusting the up and down position, and the burner angle adjusted by the controlling rod and nut. Then, adjust the transmittance to 100%.

4. Calculation method of the Measurement

Although most analysis of hydride generation atomic absorption spectrometry uses peak height as the calculation method, it sometimes uses peak area as the calculation method. Whether the peak height or the peak area is chosen, user can retrieve the data with another calculation method. However, the calculation method of average is hardly used.

5. Reading

The reading of the instrument is automatically. When all the preparations are completed, press “Start”, the hydride generation device will automatically run the procedures as following (Fig. 4-3):

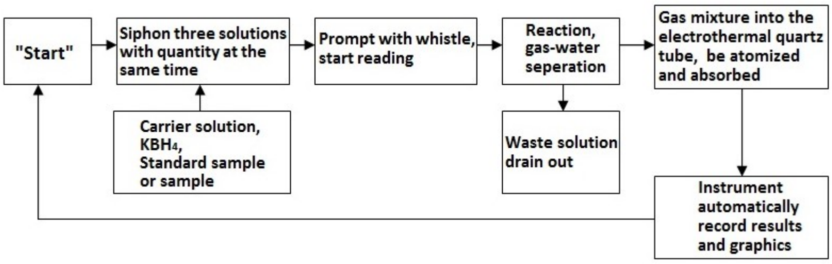


Fig. 4-3

1) System cleaning: All the pipelines will be automatically cleaned during the whole pneumatic procedure.

2) Excluding start-up and sample replacing, the whole pneumatic procedure is automatically completed by the pneumatic driver.

3) Measurement and delay time setting: The measurement time of the hydride generation atomic absorption spectrometry is usually selected in between 12 s and 15 s, so that the maximum height can be captured. The optimal result can’t be obtained if the measurement time is set within several seconds. The delay time can be between 2 s and 3 s, and with none (0 s) is also ok.

6. Static performance qualification

The static performance qualification of hydride generation atomic absorption spectrometry is mainly like the same as flame atomic absorption spectrometry. However, all the hydride elements have relevant high performance lamps. For sensitivity, detection limit and RSD improvements, here using with high performance lamp is suggested.

7. Measurement method setting

The measurement methods such as standard curve and standard addition described in operation instructions of flame atomic absorption spectrometry are all applicable to the hydride generation atomic absorption spectrometry. It is omitted here.

Chapter 5 Operation Instructions of AES

1. Atomic emission spectrometry selection

When atomic emission spectrometry is selected in the parameters setting interface, following are the operation instructions:

For atomic emission spectrometry and atomic absorption spectrometry have exactly opposite principles, the operations of atomic emission spectrometry are different to atomic absorption spectrometry. However, the parameters setting are the same.

2. Static conditions setting

1) Wavelength setting

The wavelength setting depends on the sample to be measured. For example, the characteristic wavelength of sodium is 589.0 nm, just find out the wavelength by wavelength scanning. Other operations are like the same to atomic absorption spectrometry.

2) Lamp current, negative high voltage, gain and bandwidth setting

The lamp current, negative high voltage, gain and bandwidth setting depend on the sample content. They also depend on the instrument stability, interference and sensitivity. And trials are needed before the determination.

3) Lamp position adjusting

Lamp position adjusting should be done before above conditions setting, otherwise they will be inaccuracy.

4) Transmittance adjusting

Beside the consideration of stability, sensitivity and interference, the transmittance of the standard sample with the maximum concentration also should be considered, and it should be within 100%.

3. Standard sample measurement

When the wavelength scanning interface is transferred to the standard sample measurement interface, the element lamp will be switched off automatically. And user can do the standard sample measurement.

4. Standard curve establishing

The operation of standard curve establishing is like the same as atomic absorption spectrometry.

5. Sample measurement

The operation of sample measurement is also like the same as atomic absorption spectrometry.

6. Other operations

The operation procedures such as standard addition method and output are also like the same as atomic absorption spectrometry.

Chapter 6 Operation Instructions of GFAAS

Graphite Furnace Atomic Absorption Spectrometry is the most sensitive method in the three methods of atomic absorption spectrometry. And it’s the most complicated method in the three methods with much more technology and operation requirements. Not only the measurement elements are extended, but also the measurement range of concentration is enlarged, so that the trace and ultra-trace measurements from 0.01 pg to1000 pg are available. Compared with other two methods, graphite furnace atomic absorption spectrometer is much too expensive, it needs more consumptive material, and it costs much long time and much more energy. For the method covers a broad area, and some is still in further studying, and it requires advanced technology theory and well operation skills. So, flame atomic absorption spectrometry or Hydride Generation

Atomic Absorption Spectrometry is suggested for first consideration unless graphite furnace atomic absorption spectrometry is necessary to be chosen.

However, the technology of graphite furnace is developed much more in recent years, more questions are solved excepting a long time cost and high cost for a sample measurement, and it is accepted by more users. Especially for trace and ultra-trace analysis, furnace atomic absorption spectrometry is one of the most important analyzing methods.

For the graphite furnace atomic absorption spectrometry can analyze trace and ultra-trace samples with the content between 0.01-1000 pg, there are many factors influencing the analysis. Besides the installation requirements described in chapter 2, following conditions also should be considered:

1) The sampling volume, sampling interval, and sampling position in the graphite tube have great influences on the measurement sensitivity and precision.
2) The instrument conditions such as lamp current, negative high voltage, bandwidth, and lamp position are also with important influence.
3) The graphite tube type, such as ordinary graphite tube, pyrogenic graphite tube, and platform graphite tube, also has a great influence on the measurement. Even for the same graphite tube type, it also has a great influence because of the dispersion.
4) The heating program setting, especially of the heating temperature in each stage and the internal gas supply with the atomization stage (whether on or off), are also with important influence.
5) The temperature difference of the graphite furnace also will cause measurement error.
6) Complex chemical reaction with the sample may occur in the graphite tube, and other elements in the sample may influence the measurement.

Above all, the factors that affect the analysis with graphite furnace method are extremely complex and diverse. The analyst should have a wide range of knowledge, and accumulate rich practical experience, so that to analyze with graphite furnace method accurately and reduce the measurement error.

6.1 Heating program setting

The heating temperature controlled by the computer can be from the room temperature to 3000℃, and the heating program contains four steps: drying stage, ashing stage, atomization stage, and clear stage. Both the drying and ashing stages can be divided to eight stages, and both the atomization and clear stages can be divided to two stages. For the whole steps, either ramp heating or step heating can be chosen in each stage. However, at least one stage should be chosen in each step.

1. Basic principles of heating program setting

Following are the basic principles of setting the heating program:

1) Avoid sample bumping and bubble formation.

2) Avoid the element loss before atomization.

3) Select appropriate atomization temperature.

4) Reduce the background absorption.

2. Descriptions of heating program

1) Drying stage

The purpose of this step is to make the sample to be dry without any loss.

Drying temperature setting: The principle of temperature setting is as following: The lowest temperature for drying should be lower than the vaporization temperature of about 20-30 percent, and the highest temperature should be higher than the vaporization temperature of about 20-30 percent. For example, a water solution with the ramp heating from 80 ℃ to 120 ℃, and with the step heating from 90℃ to 95℃. The latter is slightly lower than the vaporization temperature.

Drying time setting: Every liter of the sample may cost about 2~3 seconds to vaporize under around 100℃. For 10 μl sample, the standard drying time is 30 seconds. However, for the sample with deal of coexisting substance, 60 seconds drying time should be better.

Please pay attention to bumping and bubble formation

2) Ashing stage

The purpose of this step is to remove the coexisting organism and low boiling point inorganic. Then to reduce the interference of coexisting substance and smog which may bring background absorption.

Ashing temperature setting: The ashing temperature can be set from 150℃ to 1500℃, and it is usually set under the range of 400℃-700℃. The ashing temperature has impact on the background absorption and the atomic absorption sensitivity. The background absorption can be reduced with a proper ashing temperature, and so that the sensitivity can be improved. The background absorption may increase with improper ashing temperature, and the sensitivity may be reduced.

Ashing time setting: The ashing time setting rests on the sample volume. For example, the ashing time is about 30 seconds for 10μl sample. Usually, step heating is not suggested for water sample. For those samples with high viscosity, ramp heating is suggested to avoid bumping, bubbles and other abnormal phenomena.

3) Atomization stage

The purpose of this step is to form atoms, and reduce or eliminate the background.

The atomization temperature should be just right. It should not be too high, otherwise, the measurement repeatability of low temperature elements will become worse, and it's bad to the service life of the graphite furnace. And it should not be too low, otherwise, the sample can't be atomized entirely, and it will cause residues, the measurement repeatability also will become worse, and the measurement sensitivity also will be reduced.

The atomization time should be just right. If the time is too short, the absorption signal can't return to the baseline and residues will form. The time should not be too long, otherwise, it will be harmful to the service life of the graphite tube. It should be within 8 seconds. Generally, an atomization time of 3-5 seconds will be suitable.

Atomization temperature setting: Generally, it depends on the atomization characteristic of the element. For low temperature elements, such as Na, K, Pb, Cs, Ag, Au, Zn, Cd, Hg, Ga, In, Tl, As, Sb, Bi, Se, Te, Mg, Mn, and I, the atomization temperature could be no more than 2200℃. For medium temperature elements, such as Li, Sn, Ca, Cu, Sr, Rh, Pt, Be, Sm, Eu, Tm, Yb, S, Cr, Fe, Co, and Ni, the atomization temperature could be between 2000℃ and 2500℃. And for high temperature elements, such as Ba, Ru, Ir, V, Mo, U, Ti, So, Y, La, Ce, Pr, Nd, Gd, Tb, Dy, Ho, Er, Lu, Si, Ge, B, Re, and P, the atomization temperature could be no less than 2500℃. However, it is not an absolute classification method for the atomization temperature that described above. Sometimes, time controlled high power heating will be chosen, heating with a much higher power than that used for normal atomization temperature. When it reaches the atomization temperature, the system will return to the normal power supply mode to keep the graphite furnace on the atomization temperature. Above heating mode is just the controlled high power heat3, and the valid temperature range is 2500℃~3000℃.

Atomization time setting: It depends on the atomization temperature of the element. For low or medium melting point elements with the atomization temperature lower than 2600℃, the atomization time could be 3~5 seconds. For high melting point elements with the atomization temperature higher than 2600℃, the atomization time could be 4~6 seconds. The atomization time can be properly shorten, and the atomization temperature can be reduced with about 100-200 ℃ when choosing time controlled high power heat3.

Note:

a. Although the atomic absorption signal returns to the baseline, a much high absorption peak still can be existed in the clear stage, which is from the graphite tube contamination. Then, several times of "Null burning" should be done to eliminate the contamination. Otherwise, replace with a new graphite tube.

b. When the background value is large, it should be removed by ventilation with carrier gas.

c. Please reduce the sampling volume, and increase the carrier gas flow when the atomic absorption signal can’t return to the baseline, which is usually caused by a high concentration.

4) Clear stage

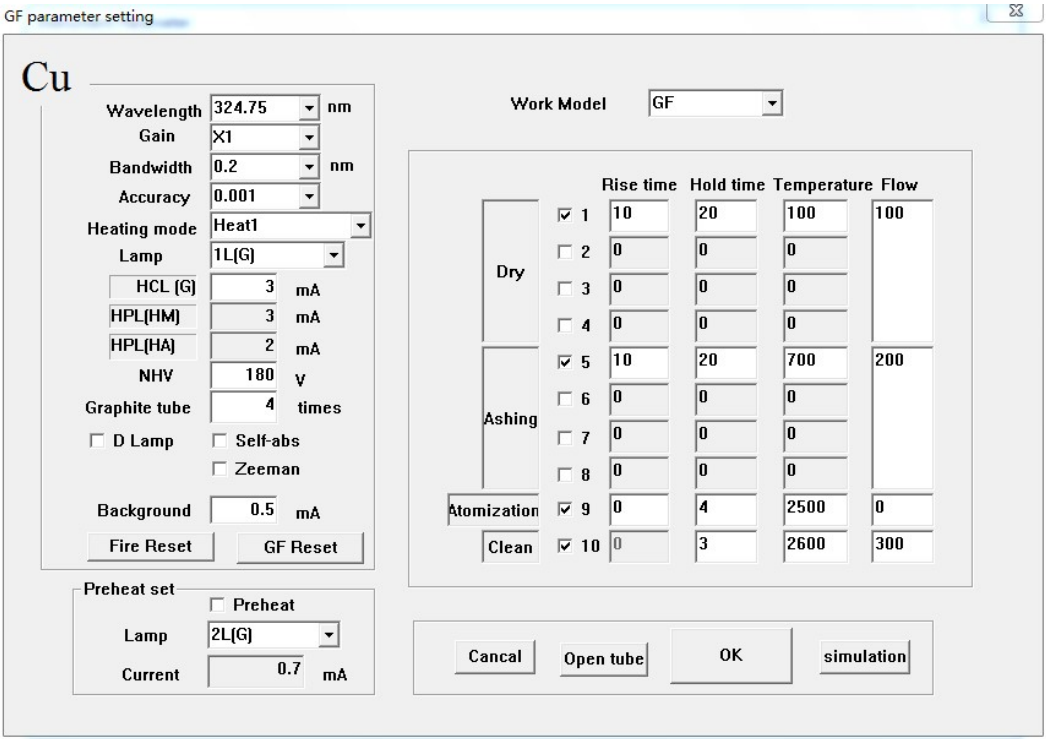
The purpose of this stage is to remove all the residues on the graphite tube.

Clear temperature setting: It could be higher than the atomization temperature with about 100-200 ℃.

Clear time setting: It depends on the contamination. A clear time of 3~5 seconds is usually used.

Note: The clear stage should be with a carrier gas, and the carrier gas flow should be high enough.

For example of the heating program setting, please refer to the parameters setting interface of the graphite furnace (Fig. 6-1).


GF parameter setting

Cu

Wavelength 324.75 nm
Gain X1
Bandwidth 0.2 nm
Accuracy 0.001
Heating mode Heat1
Lamp 1L(G)
HCL (G) 3 mA
HPL(HM) 3 mA
HPL(HA) 2 mA
NHV 180 V
Graphite tube 4 times
☐ D Lamp
☐ Self-abs
☐ Zeeman
Background 0.5 mA

Preheat set
☐ Preheat
Lamp 2L(G)
Current 0.7 mA

Work Model GF

		Rise time	Hold time	Temperature	Flow
Dry	☑ 1	10	20	100	100
	☐ 2	0	0	0	
	☐ 3	0	0	0	
	☐ 4	0	0	0	
Ashing	☑ 5	10	20	700	200
	☐ 6	0	0	0	
	☐ 7	0	0	0	
	☐ 8	0	0	0	
Atomization	☑ 9	0	4	2500	0
Clean	☑ 10	0	3	2600	300

Fig. 6-1

Take the element of lead as an example. The heating program contains four steps: Drying stage (can be divided to 8 stages, with 4 ramp heating stages and 4 step heating stages), ashing stage (can be divided to 8 stages, with 4 ramp heating stages and 4 step heating stages), atomization stage (can be divided to 2 stages, with 1 ramp heating stage and 1 step heating stage) and clear stage (with 1 step heating stage). Generally, there are 4 stages, 2 stages and 1 stage respectively for drying stage, ashing stage and atomization stage. Besides, a cooling time is necessary after completing the heating program, and there are three selections, 15s, 30s, and 45s.

Following is a simulated diagram for the heating program (Fig. 6-2).

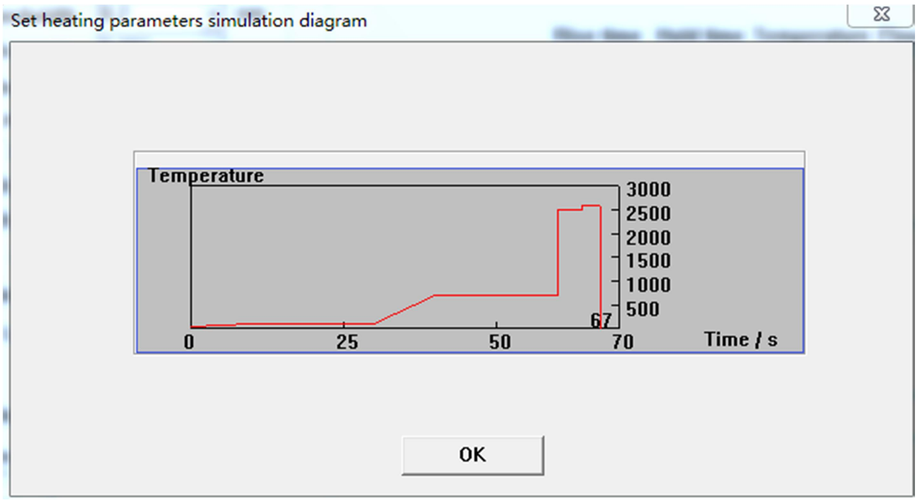


Fig. 6-2

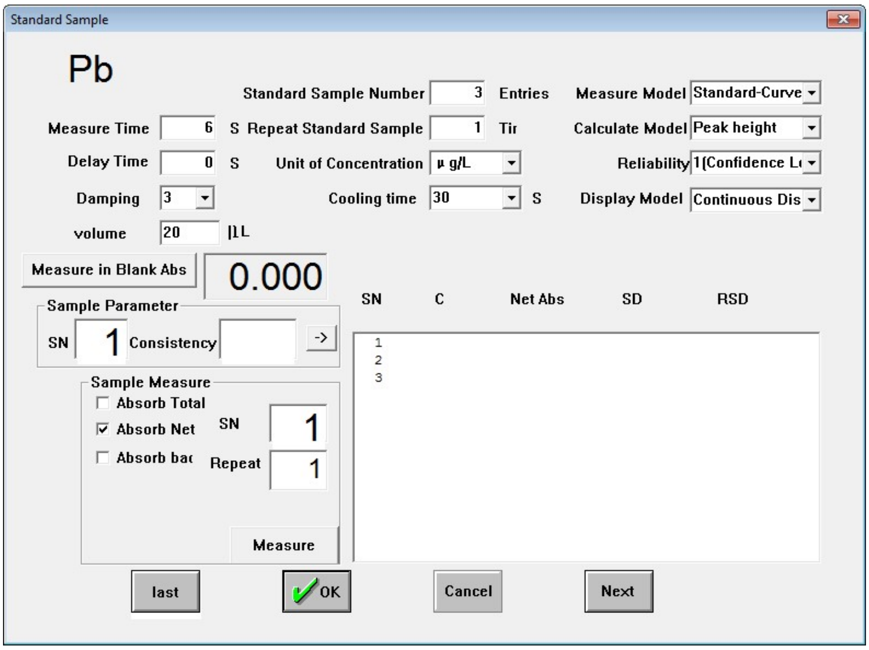
6.2 Performance Qualification

The operations for performance qualification of the graphite furnace atomic absorption spectrometer are based on the flame atomic absorption spectrometer, when the transmittance reaches to about 100%, click "Graphite furnace reset", the graphite furnace atomization device will automatically move into the light path. Adjust the instrument to obtain the maximized transmitted beam. At last, click "Auto energy" to make the transmittance to be 100%, and the performance qualification is completed.

6.3 Operation procedures

For graphite furnace atomizer is a kind of atomic absorption spectrometry, most of the analyzing conditions are the same, here only illustrate the differences for reference.

When graphite furnace atomic absorption spectrometry is selected in the parameters setting interface (Fig. 6-1), the heating program setting box will be shown in the right. Set the heating temperature, duration time, and internal gas flow according to the setting principles and methods that described in "**Heating program setting**". Then, fix up the graphite tube and click "OK", the instrument will do self-checking automatically and display the parameters setting interface (Fig. 6-1). Click "OK" again and switch to the wavelength scanning interface (Fig. 3-7) when all the requirements are met. However, this interface is without the "Ignition" button. Other operations are like the flame method. Click "Next" and switch to the standard sample setting interface. Compared to the flame method, there are several differences. The first difference is that there are three kinds of cooling time for chosen, 15s, 30s, and 45s, with 30s as the default. The second difference is the display mode of the signal, there are three selections (shown in Fig. 6-3)


Standard Sample

Pb

Standard Sample Number 3 Entries

Measure Model Standard-Curve

Measure Time 6 S

Repeat Standard Sample 1 Tir

Calculate Model Peak height

Delay Time 0 S

Unit of Concentration µ g/L

Reliability 1(Confidence L

Damping 3

Cooling time 30 S

Display Model Continuous Dis

volume 20 µL

Measure in Blank Abs 0.000

Sample Parameter

SN 1 Consistency

Sample Measure

☐ Absorb Total

☑ Absorb Net

☐ Absorb bac

SN 1

Repeat 1

SN	C	Net Abs	SD	RSD
1				
2				
3				

Fig. 6-3

a. Segment display: Display the times and signal waveforms of the whole heating program (drying, ashing, atomization and clear) for each sample.

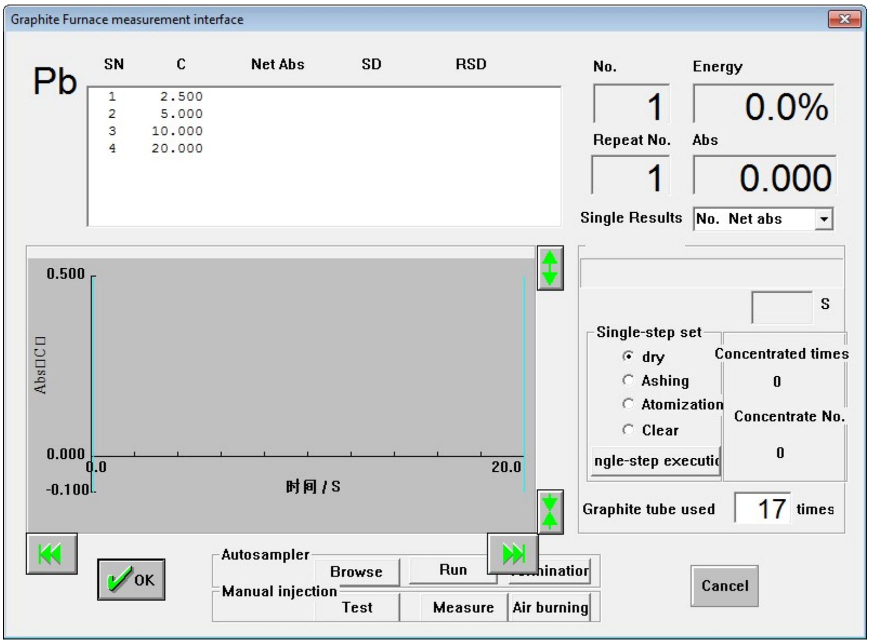
b. Continuous display: Continuously display the time and signal waveform of atomization for each sample.

c. Overlap display: Display multiple samples in the same graph, and respectively display the time and signal waveform of atomization repeatedly for each sample.

Other settings and operations are like the same as the flame method.

Click “Blank” and switch to the graphite furnace measurement interface (Fig. 6-4). If a new graphite tube is used, several times of “Null burning” should be done until the absorbance is close to zero. User can click “Test” to check whether the program is run with the settings or not. In the “Single-step set” pane, whether drying, or ashing, or atomization, or clear, once be chosen, and the button “Single-step execution” is clicked, the system will run the single step. Up to 20 times of “Concentrated times” which is used for sample concentration can be set. Either standard sample or sample to be measured, once the concentration procedure is selected, all the concentrated times should be run before measurement.

Click “Measure” after adding the sample into the graphite tube, the whole heating program will be run, and the measurement result and the corresponding waveform will be given at last.



SN	C	Net Abs	SD	RSD
1	2.500			
2	5.000			
3	10.000			
4	20.000			

No. 1

Energy 0.0%

Repeat No. 1

Abs 0.000

Single Results No. Net abs

● dry
○ Ashing
○ Atomization
○ Clear

Concentrated times 0

Concentrate No. 0

Graphite tube used 17 times

Fig. 6-4

Other operations are the same as the flame method.

Power off: Disconnect the USB connection first. Then, respectively switch off the power of the graphite furnace and the instrument. After that, turn off the computer. At last, cut off the water and the gas, and switch off the total power supply.

Chapter 7 Maintenance

7.1 Instrument Maintenance

1. Periodic inspection

Whether the instrument is used or not, especially without using for a long time, periodic inspection should be done. Generally, six months a time is necessary. The inspection includes following contents:

1) Complete inspection

Use the hollow cathode lamp of copper to check the whole instrument completely. Set the wavelength at 324.7nm, with the lamp current of 3mA and the bandwidth of 0.2nm. When the transmittance reaches 100%, record the negative high voltage, and save these conditions to certain file for later reference. Of cause, it should be on premise of the conditions being all right, and the lamp position should be adjusted right, the wavelength should be at the peak wavelength. Compare with each negative high voltage in each measurement, various problems can be explained. Such as the transmittance variety between the first lens and the second lens, the element lamp lifetime cost, whether the circuit of the instrument is abnormal or not, whether the monochromator works all right or not. Periodically run the instrument with proper time can keep the electric circuit dry.

2) Atomization system inspection

Use the hollow cathode lamp of copper to check the atomization system, with the same static conditions as above, block the light path when the burner is cold, and record the value. Compared with the values and to explain some problems, such as whether the measurement sensitivity is reduced or not, whether the burner is corroded or not, whether there is a residue in the nebulizer chamber or not, whether the performance of the nebulizer is ok or not.

3) Drying cylinder inspection

Periodically check the silica gel in the drying cylinder. Normally, they should be blue and transparent. If they become red, replace them with new dried ones timely, and the operation should be done without electricity.

4) Hollow cathode lamp inspection

Generally, if the hollow cathode lamp is not used for a long time, to avoid spontaneous damage, it should be timely lighted six months a time.

2. Routine maintenance

1) After completing the sample measurement, spray with the distilled water about 5-15 minutes (the time length depends on the sample concentration). If the instrument will not be used for several days or a longer time, run “Null burning” about 10-15 minutes to avoid the corrosion of the burner and the nebulizer chamber.

2) Regularly clean the burner and the nebulizer chamber to reduce the risk of corrosion and avoid the memory effect. Regularly check the mechanical system with lifting up and down, moving back and forth to prevent corrosion. Regularly check the gas pipeline to avoid the fuel gas leakage. Regularly wipe off the dust and sputtering liquid on the first and second lens, just with the mixture of pure alcohol and ether (1:1), then, gently scrub with polyester cotton. To prevent the lens from damage, please don’t touch the lens with a hard object

7.2 Maintenance and Troubleshooting of the Hydride Generation Device

1. Water injection

Clamp the water inlet tube, remove the plug, fill the syringe with distilled water, and connect the syringe to the water inlet tube. Inject the water until it reaches the highest level that marked with a blue line before the whistle. Clamp the water inlet tube, remove the syringe, and block the inlet with the plug. The water will evaporate after a long term use, so it should be added with above method when the liquid level is lower than the marked line.

2. Electrothermal quartz tube cleaning

Some milk-white salt spray deposition will be on the inner surface of the absorption tube after a long term use, which will react with hydrogen and generate hydrogen radical, take the hydrogen from the hydride and be atomized, and cause the measurement sensitivity decline. So cleaning the absorption tube should be done regularly. User can use hydrofluoric acid to clean the absorption tube. Place the absorption tube vertically, and block both ports of the bottom and the branch with plastic plugs, then, pour concentrated hydrofluoric acid into the tube from the upper port, pour the liquid out after holding 15 minutes. Then, rinse with water. Above method can be used once only, and next time, it should be replaced with a new absorption tube.

Note: Please be carefully and don’t drop the acid onto the heating cord. Otherwise, the insulation layer will be dissolved and causes short circuit.

3. Potassium borohydride no inhalation

   Remove the No. 0 capillary tube in the lower left corner at rear of the hydride generation device, inject water to the capillary tube with a syringe to keep it unobstructed, then, fix it back. If it is still obstructed, gently heat with a small fire and inject water so that it will be unobstructed. Otherwise, replace with a new capillary tube.

4. Carrier liquid no inhalation

   The No. 1 capillary tube is for carrier liquid. To keep it unobstructed, the operation is like the same as the No. 0 capillary tube.

5. No back-blowing after sampling tube filling with sample solution

   Open the cover of the hydride generation device, find out the No. 2 capillary tube, make it unobstructed and fix it back.

6. Sample and solutions inhalations ok, but no absorption signal

   Check the mixed gas tube, and keep it unobstructed with gently pull. If there is some water on the inner wall, blow it dry with air. The liquid level of gas-liquid separator should be above the lower marking line.

7. Start button can’t return to its position after reading whistling

   Check the pipeline, make sure that the vent-pipe is unobstructed and the pressure is adequate.

8. High baseline and large blank value

   Sampling with high concentration samples, may cause contaminations to the pipeline of the hydride generation device, and cause high baseline and large blank value. If that occurs, don’t do automatically zero, just increase the carrier gas flow to blow away the contaminations on the pipe wall until the blank value return to normal. Another way, directly connect the compressed air to the mixed gas outlet pipe to blow in reverse until the blank value return to normal, and the waste tube should be clamped first.

9. Measurement sensitivity too high

   If the measurement sensitivity is too high, user can increase the carrier gas flow, or dilute the sample. If it still can’t satisfy the requirement, please contact us.

10. Open flame or explosion in the graphite tube

Phenomena of the open flame and explosion are the reflections of impure carrier gas (nitrogen or argon) with too much oxygen. Please replace with high purity carrier gas.

7.3 Maintenance of the Graphite Furnace Device

As the graphite furnace method is mainly for trace and ultra trace measurement, user should pay more attention to the maintenance of the furnace body.

1. Graphite tube

The graphite tube is the core of the furnace body, and it has a direct impact on the measurement results. Generally, for the first using, several times of “Null burning” should be done until the absorbance is close to zero. Contaminations may exist after a period of time unused, to obtain accurate measurement results, several times of “Null burning” also should be done before reuse.

Besides, pay attention to the usage frequency. Generally, a temperature calibration is suggested after about 50-80 times use. If the measurement repeatability becomes poor and the sensitivity varies greatly, it may be the service life of the graphite tube exhausted, and it’s time to replace with a new one.

2. Graphite cone

The graphite cone is an intermediary to ensure the reliable contact between the graphite tube and the electrodes. The contact quality of the graphite cone directly affects the graphite tube for that whether the set temperature can be steadily arrived or not. Especially for those medium and high temperature elements, more attention should be paid. The graphite cone may be contaminated and cause the poor measurement repeatability and varied sensitivity. In that case, user should remove the graphite tube from the furnace body and clean the graphite tube with polyester cotton that dipped in the mixture of alcohol and ether (1:1).

3. Shielded quartz window

The shielded quartz window is used for sealing the protection gas. For the light beam from the hollow cathode lamp should pass though it, it should be with high transmittance. However, its inner surface may be polluted by the graphite powder from the graphite tube and the sputtering liquid from the sample. Once the transmittance becoming low and can’t return to the initial level, it may be the pollution problem of the shielded quartz window. Remove it from the furnace body and clean it with polyester cotton dipped in the mixture of pure alcohol and ether (1:1). Periodic cleaning also can be done if necessary.

7.4 Notices

To prolong the service life of the instrument, for safety usage and to keep the best performance of the instrument, user should pay attention to following notices:

1. The monochromator of the instrument has been adjusted well before delivery. To keep its good performance,

please don't disassemble it without authorization. Otherwise, any consequence brought about due to this should be charged by the user.

2. To avoid unnecessary lost, please don't plug in or pull out the power plug during the instrument running.
3. If the instrument is not commonly used, and user want to keep the wavelength set in the parameter setting interface, user can save the wavelength parameter under the system reset. When the instrument is commonly used, and the wavelength is not accurate, the system reset should be done. After that, do wavelength scanning and make sure that the wavelength is accurate.
4. A water seal is needed for the flame method. User should connect the waste tube and add water to the waste tube before measurement. Otherwise, the instrument may run unsteadily, even cause an accident of backfire.
5. The instrument provides automatic protection of the gas system, so that user needn't to be scared when a power failure occurs while ignition and sample atomizing. However, to avoid the fuel gas leakage when the power is suddenly recovered, user should turn off the needle valve of the fuel gas first. Then, switch off the instrument power and the computer power. It also means that the operator should not leave the instrument as long as the ignition. And for safety consideration, user should pay attention to such an exceptional case.
6. When hydride generation method is chosen, user should check the liquid level of the gas-liquid separator tube whether the requirement is met or not, otherwise, water injection should be done first.
7. Periodic replacement of the silica gel is needed when the instrument lays idle.

Any questions or comments about the documentation for this product, please contact us:

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