Operating Manual

AA 7800

Atomic Absorption Spectrometer

Safety class and type:

Safety class of the AAS: class I

Appearance safety class: IP20

Statement:

The relevant information in this manual is subject to change without notice.

This manual strives to be accurate and comprehensive. Please forgive me for any errors or omissions.

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Instructions for safe operation:

Prompt label:

The prompt flag prompts the user to pay attention. Misoperation will lead to data loss or destruction.

Please use it as required. Misoperation is strictly prohibited.

Warning label:

The warning label contains all possible dangerous situations. If you do not follow the correct steps during program operation, you may cause accidental injury to operators. Please operate with caution!

Tips:

Please read this manual carefully before using the atomic absorption instrument.

The atomic absorption spectrophotometer produced by our company is a safe and reliable instrument; If users use it properly, they will get accurate, true and reliable analysis data. Failure to operate in accordance with the instrument instructions may cause damage to the instrument and distortion of data. The company is not responsible for the loss of samples or personal safety accidents caused by the misuse of instruments, consumables or accessories.

1、Atomic absorption spectrophotometer is an analysis and test equipment, and the operator shall receive professional training before operation.

2、 Do not install or use this instrument for anything other than analytical testing.

3、 The installation and service of instruments or parts, including the installation and service of accessories, shall be performed by trained and authorized installation and commissioning personnel.

4、 The computer connected atomic absorption spectrophotometer shall be specially used for the operation of the instrument and the processing and processing of analytical data. The third-party software executed in the process of analysis and measurement may cause data loss.

Contents

SAFETY INSTRUCTION

I. Summary

Please read this section before using the AAS in order to ensure safe operation. Comply with all safety instructions in this manual and pay close attention to all messages which noticed by AAS software.

Also follow the safety instruction of accessories product manual by supply manufacturers (PC, Printer). It is particularly important to comply with the safety instructions on the labels to handle, storage and disposal the reagents.

Atomic absorption spectrometer uses compressed gas and high voltages may produce radiant and hazardous waste include corrosive fluid and flammable liquid. Incorrect operation may cause serious consequence. This manual contain instruction to guild users to operating instrument safely. To ensure safe operation, install, disassemble, or move instrument should be under professional supervising.

The shell of instruments and accessories must not be removed by the operator, except for the routine maintenance only performed by Spectrum instrument engineer. Read these safety guilds thoroughly before attempting to operate the instrument and always operate the spectrometer under safety instruction.

The HGA uses interlocks and shells are designed to prevent accidental of potential hazards, therefore interlock or shells of instrument should not be removed without permission. Fumes generated by the device can be hazardous and must be extracted to outside air. Routinely check the exhauster to ensure it functioning well and switch the exhauster on before conduct analysis on instrument.

Purpose

The AAS is used for atomic absorption and atomic emission analysis. Do not use for other purpose。

Regulations

Comply local safety regulation such as work safety regulation, etc.

Operator must be familiar with the content of this manual; only skilled and trained personnel shall operate instrument.

II. Warning and Caution Sign

Warning Sign

The following is a list of symbols that appear in this manual in conjunction with warnings on the instrument. The meanings of the symbols that may appear alongside warnings in the documentation or on the instrument itself are as follows:

Danger! This sign indicated that there is potential of danger, false operation may cause damage or injury.

Danger of electric, shock if touched!

Danger! Hot surface! The hot surface can cause hurts if touched

III. Electrical Hazards

Electric shock!

The electronic control module of this instrument contains electrical circuits and components operating under high voltage. Contact with these components can cause electric shock result in injury or even death. Instrument panel or shell should only be removed by Spectrum service engineers. Use wrong supply power, voltage or connect to incorrect outlet which lack of appropriate for grounding will cause fire or electric shock danger result in serious injury, death or damage to instrument.

Please use a third-line socket with grounding

- The installation must comply with local safety regulations.

- Ensure the voltage is correctly set before connecting to the instrument.

Lethal voltages may exist in instrument components! The instrument should connect to a properly power socket of grounding to ensure safety, while using extension line, the protective conductor will fail. The AAS and its attached device such as computer or printer should all connect to socket with grounding. The instrument requires shutting down and unplugging before any maintenance. The instrument back shell's removal should only be operated by Spectrum engineer or skilled professionals.

Do not attempt to override or disable interlocks. Failure to comply this warning can result in death or serious injury.

Must always unplug the mains power inputs when direct access to the electronic component.

Switching off instrument during emergency!

In case of an emergency, switch off the AAS and unplug the instrument. Disconnect all device and switch of the circuit breaker. Ensure circuit breaker is easy to reach for operators.

Caution! Disconnect and switch off the PC in an emergency has a risk of data loss and damage to the operating system!

IV. **Compressed Gases Hazards**

Compressed gas usually storage in gas cylinders. Gas cylinders should be placed by strictly following safety instruction.

Compressed gases can create a hazard if rapidly depressurized or leaking into the air. Combustible gases, such as ethyne (C_2H_2) or propane (C_3H_8), can result in a fire incident. Others such as N_2O,can result in an anesthetic or oxygen-deficient atmosphere.

The standard oxidant for flame AAS is air, which can be supplied from pressurized cylinders or compressors. Pure oxygen must not be used as an oxidant in flame AAS

Gas cylinders must be handled and stored strictly according to local regulations and safety measures.

They must be used and stored exclusively in a vertical position and must be fixed in an approved cylinder stand. The room in which cylinders are used and stored must be adequately ventilated to prevent accumulation of gases in case of small leaks.

Cylinders with compressed gases must be kept at moderate temperatures which should not exceed the usual working temperature of a laboratory at max. 35°C.

All cylinders and tubes connect to the cylinder or instrument must be clearly labeled, to ensure the gas supply correctly. All gas connectors and tubes need to be correctly installed. Any damaged tubes must not be used. Routinely test leak with soap bubble solution (e.g. once per week) and required immediately when there is leak found. Keep ventilate after changing the gas cylinder.

Cylinders contain burnable gases should equip with threaded fittings in anti-clockwise direction, non-burnable gases use clockwise-directed threads.

Air supplied from a compressor must be dried before being supplied to the gas-box of the instrument. Moisture can damage the components of the gas-box. All gas supplies should only be opened when the instrument is in operation!

- **Ethyne, C2h2 (Acetylene):**

Ethyne should be used at pressures below 0.15 MPa (~22 psi). The AAS requires an inlet pressure of 0.09 ± 0.02MPa for ethyne.

Ethyne may explode if direct contact with noble metals such as e.g. silver, copper etc. or subject contain grease. Copper or copper-rich brass tubes or fittings must not be used for transporting ethyne.

Ethyne is usually supplied in cylinders containing acetone. Only these pressurized gases must be used for operating AAS. The pressure in the cylinder is not allowed to fall below 0.50MPa (~72.5 psi) as acetone may be carried into the gas box of the spectrometer. This may cause corrosion and malfunction of the system .

The rate of gas drawn from the cylinder should be moderate to prevent acetone from being carried with the ethyne. The ethyne used should at least be of "instrument grade" with a purity of 99.5%.

- Compressed Air (Flame):

Never use oxygen-enriched air or pure oxygen as the oxidant. This will result in an immediate flash back in the burner system!

- N2O (Nitrous Oxide):

Nitrous oxide flames have an extremely high burning velocity. For nitrous oxide flames, it is most importance to use approved burner head. Oxidant flow conditions at the nebulizer must not be changed while operating the nitrous oxide flame! Decompression of nitrous oxide gas will cause strong cooling at the regulator. Incoming piping, screwed joints and pressure reducers for nitrous oxide must be kept free of grease. In order to avoid condensation of moisture or freezing and malfunction of the regulator valve, the regulator should be equipped with a heater.

- Argon

An inert gas is obligatory for operation of the graphite furnace. It is recommended to use only Ar as an inert gas connected to the 'Normal' gas inlet。

- Air (Graphite Furnace)

  Air may be used at the alternate gas inlet. In GF-AAS it is usually supplied from a pressurized cylinder.

- Water

  During installation of Furnace AAS, the service engineer will connect the unit for the cooling water supply. The pressure of the cooling unit should be between 0.09 MPa and 0.11 MPa (~27 psi to 30psi).

V. Flame Operation

- Heat, Fumes And Vapors

  Flames used in AAS generate heat, fumes and vapors. These can be hazardous to people. The instrument must be positioned underneath a proper extraction system with a flow of at least 10 m3/ min.The exhauster must be vented to the outside air and has to be in accordance with local regulations for ventilation and fire prevention. The hood, ducting parts and joints must be heat-proof. The motor of the exhauster is not allowed to exceed its maximum operating temperature. The exhauster must be switched on before the flame is ignited. The system must be checked regularly for proper operation,e.g. by watching the extraction of smoke.

- Nebulizer, Mixing Chamber, Liquid Trap And Burner:

  Incorrectly assembled and mounted nebulizers can create fire and explosion hazards. Nebulizers must be correctly mounted and fitted to the mixing chamber before igniting the flame. Nebulizers must be adjusted so that there is always a negative pressure towards the sampling capillary. The nebulizer must be optimized for best flow conditions only with air-ethyne flames. The nebulizer settings must not be changed while a nitrous-oxide flame is in operation. Removal of the nebulizer from the mixing chamber and all service actions, e.g. cleaning of the nebulizer capillary, must not be executed when the flame is in operation. The proper fit of the nebulizer in the mixing chamber and the absence of leaks should be checked on a regular basis。

✧ Liquid Trap:

The liquid trap is an integral part of the mixing chamber. It is equipped with a sensor which allows the flame to be ignited only when the trap is filled with solvent up to a safe level. The proper function of the liquid trap is essential to protect the work environment as well as the waste bottle from being filled with burnable or explosive gas mixtures. The liquid trap interlock is an essential part to minimize the risk of gas leaks through the drain. It must never be bypassed.

The liquid trap must be filled with the same solvent being used for the measurement solutions. Safe operating conditions, however, can only be assured for solvents having a specific gravity of higher than 0.75.

The liquid trap is drained by tubing which transports the liquid waste to a suitable vessel (a sturdy Teflon or PP waste bottle with a stable base and an enough volume to hold the amount of waste liquid produced in 8 hours of experimenting, which would be at least 4 liters). The end of the tubing must remain above the liquid waste in the vessel. The drain vessel should be made of a plastic material which is resistant against strongly oxidizing acids and organic solvent.

✧ Burners

Air-ethyne flames reach temperatures of up to 2300°C, Nitrous oxide-ethyne flames may reach temperatures of up to 2900°C. The respective burner heads may be at 250°C-400°C after operating for some time. Whenever operator handle them have to wear heat-protective gloves. Spectrum Instruments provides a burner head with a 100-mm slot for air-ethyne flames and a burner head with a 50-mm slot for N2O-ethyne and air-ethyne flames. Always use the correct burner. Air-ethyne burners will cause an immediate flashback if used with N2O-ethyne. Burner and instrument are equipped with interlocks which recognize the type of burner head or the absence of a burner head. Never try to remove these interlocks.

Burner heads may block if used for a long time or the solutions for measurement contain higher amounts of salts or organic matter. To minimize burner blockage, the burner slot must be regularly cleaned. Blockage of the burner head is more noticed in N2O- ethyne flames. Spectrum Instruments offers an automatic cleaning device for 50mm burner heads. This “scraper” must be activated when N2O-ethyne flames are used. Severely blocked burner heads can cause flashbacks and explosion or fire hazards.

Only the automatic cleaning device should be applied for cleaning the burner head when the flame is burning. If the burner slot is cleaned with other tools, the flame should be turned off.

- Damaged burners are a safety hazard and must not be used.

- In general, flames should not be operated unattended

- Flashbacks

Flashbacks are rare in modern flame AAS. If they occur, however, the bang and the possible damage of some of the parts, may startle the users and cause them to discontinue nitrous-oxide flame operation. The following list is a guideline to avoid flashbacks completely or at least minimize them in daily laboratory routine.

In most cases flashbacks will occur if the flow rate of the burning gas mixture is slower than the velocity of the flame. Therefore, most flashbacks will be experienced when operating N2O-ethyne flames! Avoid using salts or organic matter to minimize blockage of the burner head slot. Sediment will gradually change the shape of the flame and cause turbulences, which may eventually cause flashbacks. In rare cases, glowing carbon particles formed around the burner head slot may fall inside and ignite the gas mixture within the mixing chamber. The burner slot must be routinely cleaned (see maintenance, chapter 5).

Blockage of the burner head will occur much more rapidly in N2O- ethyne flames situations which liquid contain very high solid content or organic solvents will cause flashback. In all these cases the aspiration rate of the burner should be reduced to the required minimum.

The integrity of the mechanical dimensions of the burner head slot must be ensured. Expended slots by frequent use or cleaning will result in reduced burning gas velocity, thus increasing the risk of flashbacks.

The spray chamber, liquid trap and drain tubing must be cleaned on a regular basis. Any blockage of the drain, or sludge in the mixing chamber, have the risk of changing the gas flow conditions and thus provoke a flashback.

The automatic gas box of the AAS generally ensures proper gas flow conditions. In case of pressure drops the flame is usually extinguished automatically. Nevertheless, the proper status of the gas cylinders and reducer valves must be checked before the flame is ignited.

VI. Chemical Hazards

The instruments accessory may involve the use of materials which are toxic, highly corrosive or hazardous. Improper use of such materials can cause serious personal injury.

Always ensure following laboratory safety practices, handling and disposal of such materials are

strictly observed. These safety practices should include the wearing of appropriate safety clothing.

Before attempting any work on the sample compartment area, you must know exactly what matrix of solutions has been running through the instrument and particularly what types of solvents are involved. Always check that the auto- sampler has been thoroughly flushed out with distilled water and that the contents of the drain or waste container are known and properly disposed before touching any part. Always wash your hands and gloves thoroughly and avoid contact between hands and eyes during servicing of the instrument.

The preparation of samples for element determinations with AAS usually requires the use of chemicals and solvents. Only trained professionals can handle reagents, samples and standard solvents, and operate the measurement device and its accessories. Flammable solvents introduced into a flame AAS or near a burning flame can create explosion which can result in death, or severe injury. Desolation of samples or standards within flammable solvent require specifically trained and experienced personnel All safety practices must be strictly followed.

The following recommendations are risks with flammable solvents:

✓ Reduce the amount of flammable solvents to the minimum volume required for the analytical task.

✓ Organic solvents with a specific gravity less than 0.75 must never be used.

✓ Flasks contain flammable solvents should never be near flame and always be covered up

✓ Avoid cross contamination of nebulizer capillary.

✓ All tubing, gaskets, vents and bottles should be solvent-resistant.

✓ The general plastic laboratory tube and containers are often not suitable for organic solvents.

✓ Glass containers should not be used in order to avoid glass splinters in case of an explosion.

✓ After accumulating large volume of flammable solvents, the wasted flask should be emptied and properly disposal.

✓ Do not add strongly oxidizing substances, e.g. nitric acid in order to reduce organic remain volatilize.

The operator should be much careful with selecting substances during analysis process, especially substances with radioactive, nfectious, poisonous, corrosive, combustible and explosive. When handling hazardous substances, local safety codes and guidelines must be observed.

Warnings on the labels must always be observed and only use containers which clearly labeled. Always use protective goggles and rubber glove.

The AAS must be operated under a laboratory exhaust hood (ozone, wasted gas created by burned sample, poisonous and combustible by-products from sample preparation processes must be properly disposed). Hydrofluoric acid must be carried out in an exhaust chamber and wear protective equipment such as rubber aprons, gloves and face masks.

Biological samples must be handled according to local infectious material guidelines During measuring cyanide-containing material, ensure that prussic acid cannot be generated in the waste.

Warning!

Sodium borohydride (NaBH4) is highly corrosive and hygroscopic which is extremely aggressive in solvent. Avoid dripping and spilling of reductant. Ensure that all residue liquid from the mixing chamber (siphon) and the auto-sampler is discharged into the waste bottles. The operator is responsible for ensuring that waste materials, e.g., sample residues, drained cooling agent or filter residues from the compressor, are disposed according to local regulations.

List of hazardous organic solvents

Organic solvents are often hazardous to health. They are usually highly flammable and often exhibit a high vapor pressure at room temperature. They must be handled with special care, especially near the AAS burner unit. Typical organic solvents are e.g.

- Methyl isobutyl ketone (MIBK): Highly volatile, noxious- smelling
- Toluol: Dangerous to health.
- Kerosene: Flammable, low vapor pressure.
- Methanol, ethanol, propanol: Combustible, partly dangerous to health.
- Tetrahydrofuran (THF): Highly volatile, easily ignited, dissolves polyethylene and polystyrene.

Not all the organic solvents are contained within the list, beware that any use of unknown or uncertainty organic fluid should contact manufacturer to ensure its safety

Cleaning and decontamination measures：

The operator must perform cleaning while the instrument (external or internal) is contaminated by danger or corrosive substances. Spots, drops or larger spillages should be removed and cleaned using an absorbent material such as cotton wool, laboratory wiping cloths or cellulose. The affected areas are then to be wiped with a detergent. Please contact manufacturer before attempting any cleaning method which supplied by third party to prevent any damage to instrument.

VII. UV - Radiation Emitted By Lamps And Flames

Line-specific lamps in AA (mainly hollow-cathode lamps), deuterium lamps and flames emit a wide ultraviolet radiation spectrum. This can cause damage to the eyes and skin.

It is therefore mandatory to wear protective safety glasses which conform to the relevant standards.

Light from hollow-cathode lamps, D2 lamps and the graphite furnace during a firing cycle should never be viewed directly. Even under these conditions never look the heating furnace without protective glasses!

VIII. Electromagnetic Compatibility EN55011/CISPR11

This Spectrum Instruments Spectrometer and the original Spectrum Instrument accessories comply CISPR11, Group 1, Class A as radiation professional equipment. Class A equipment is suitable for use in all establishments except low voltage power supply network.

Changes or modifications to all electrical circuits or devices not approved by Spectrum Instruments could nullify the electromagnetic compatibility and function of the instrument.

- High Temperatures

High temperatures occur when flame, graphite or chemical vapor generator is operated. The integrity of cooling system should be observed while perform operation Do not touch the hot components directly during or after a measurement. Perform maintenance and change components only after an adequate cooling period. Keep all combustible material away from the graphite furnace.

- Ventilation

Make sure that the ventilation fittings for the AAS and related auxiliary devices are in proper working condition. Covered vents or ventilation slits may cause overheating of the device and thus may cause damage to it.

- Cleaning And Maintenance

Service, maintenance and repair work on the AAS must only be carried out by service engineers from Spectrum Instruments, or by technicians authorized by the manufacturer. If these requirements are not followed, there will be a serious risk of misalignment or damage to the device.

The exterior of the AAS can be cleaned with a damp, non- dripping cloth. When cleaning the interior of the AAS, the operator is required to take safety precautions, especially in terms of contaminated and infectious materials.

- Sensitive Electronics

Never connect and disconnect electrical components of the AAS unless they are switched off.

Chapter 1 Instrument installation

1.1 SPECIFICATIONS

- Techniques

- Flame technique in single or double beam operation without background correction.
- Flame technique in single or double-beam operation with deuterium background correction.
- Flame technique in single beam operation with Smith-Hieftje background correction.
- Furnace technique in single beam operation without background correction.
- Furnace technique in single beam operation with deuterium background correction.
- Furnace technique in single beam operation with Smith-Hieftje background correction.
- Chemical vapor generation and mercury cold vapor technique in single-beam operation with or
- without deuterium background correction.

- Photometer

Assembly	Czerny-Turner monochromator with high performance 1800 lines/mm grating. Automatic control of wavelength and slit setting
Wavelength Range	185-900 nm
Slit width	0.1nm, 0.2nm, 0.4nm，0.7nm, 1.4nm，2.0nm

- Turret For Hollow Cathode Lamps

PC-controlled 4/8-lamp turret for fully automated operation. Stabilized power supply unit for simultaneous supply of 4/8 lamps in operation and/or pre-heating mode.

- Hollow Cathode Lamps (HCL)

Hollow Cathode lamps for about 68 elements. Software controlled selection of operating parameters.Lamp current settings from 2 mA-20 mA.

- Deuterium Lamp

D2 arc discharge lamp. Lamp current automatically adjusted for wavelength dependent energy match.Mean current: 150 mA.

- Absorbance Read Out And Display

Total absorbance, specific absorbance, background absorbance

- Display Modes

Absorbance	-0.1000 - 3.0000 A
Concentration	Range: 0.00001 – 99999

- Calculation method

Signal Evaluation	Mean value; Peak Height (Maximum value of the Absorbance), Peak area (Time-integrated Absorbance)
Replicates	1-30

Integration time	0 -104s
Read delay	0 -100 s
Read out units	Absorbance, Concentration
Number of decimals	4
Concentration units	Weight/volume; weight/weight; ppm, ppb, ppt.
Results display window	Data results, Calibration, Real-time atomization absorbance signal

- Calibration

Calibration techniques	Standard calibration; Method of addition; Method of addition calibration
Fit reference curve	Linear; linear through zero; non-linear; non-linear through zero
Number of standards	1 - 20
Number of addition con-centrations	1 - 20
Recalibration	Two-point recalibration with display of the recalibration factor

- Power Supply

Line voltage and frequency	200 V AC -240 V AC 50 Hz /60 Hz
Power consumption	Base instrument: 6000 VA Base instrument, PC, monitor, auto-sampler: 250VA
Safety class	I
Safety type	IP 20

Do not connect further components aside from PC and monitor to the output socket of the spectrometer. The maximum allowed current may be exceeded.

- Instrument Fuses

Voltage	Value	Function
220v	5A	Power supply fuse

- Environmental Conditions

Corrosion protection	Instrument and accessories are corrosion protected; still the system shall be protected from highly oxidative liquids and fumes
Working temperature	10°C - 30°C
Humidity	Max. 88% at +30°C

- Dimensions And Weights

Mass	90Kg
Dimensions (D x W x H)	830 mm ×650 mm × 560 mm

- Types Of Flames

Ethyne/air	One-slit burner 100 mm(standard) One-slit burner 50 mm(standard)
Ethyne/nitrous oxide	One-slit burner 50 mm (optional)

- Oxidant

Compressed air and N2O	Inlet pressure:290 KPa - 310 KPa (~42.1 psi - 44.9 psi)
Nebulizer flow:	
Air	4 L/min - 6 L/min
N2O (Nitrous Oxide)	
Additional oxidant	
Air	16steps: 0-7.5 L/min
N2O (Nitrous Oxide)	16steps: 0-7.5 L/min

- Fuel Gas

Ethyne (acetylene)	Inlet pressure：0.9±0.02Mpa Flow：0.9 - 6.3 L/min

- Nebulizer

Type	Pneumatic nebulizer with Pt//Ir capillary and Ceram bead(optional)
Nebulizer flow	4 L/min- 6 L/min

- Automatic Burner

Height	5 mm -25mm
Rotation	0° - 90 °

- Safety Circuits

Safety control functions	Burner and burner type Fuel and oxidant gas pressure Siphon level Flame presence

- Flame Auto-Sampler（optional）

Sample tray	90
Sample positions	80 (15mL)
Special positions	8 (50mL)
Power supply	By connection to AA- spectrometer
Mass	6kg

- Air Compressor

Power supply	（220±22）V, 50 Hz or 60 Hz
Inlet pressure:	0.3±0.01 MPa
Required rate of gas supply:	Max：800 L/h

- Furnace Auto-Sampler （optional）

Sample tray	100
Sample positions	85(2mL)
Special positions	4 (7mL)
Power supply	By connection to AA- spectrometer
Mass	5Kg

- Cooling Unit（optional）

Power supply	220V, 50 Hz - 60 Hz
Working pressure range	0.1±0.01MPa
Recommended Temperature range	18°C–30 °C

- Argon Gas

Argon Gas:	0.3±0.01MPa

- Air Gas

Air Gas：	0.2±0.01MPa

- Graphite Tubes

Types Of Tubes:

Pyrolytic graphite coated high density graphite tubes with Omega platform

Pyrolytic graphite coated high density graphite tubes for wall atomization

- Safety Circuits

Safety control functions	Contact temperature
	Tube status
	Water presence
	Argon presence
	Presence of alternate gas (if relevant)

1.2 INSTALLATION

Requirement:

- Requirements: Please consult the document “Installation Requirements and Announcement of Readiness for Installation, the AAS, which you have received with the shipment of the instrument and its accessories.

- The laboratory must be prepared according to the instructions in this document before the installation and startup of your Spectrum Instruments AAS.

- Operating instructions for all Spectrum Instruments Accessories are provided in the manuals included with the respective devices.

- Installation and operation of the “RGWin-AAS” software is included in this manual.

- The spectrometer and its accessories must only be assembled, installed and repaired by service engineers from Spectrum Instruments or by technical personnel authorized by Instruments.

■ Assembly and Installation

The instrument and its accessories need to be unpacked for installation. Enough space in the laboratory is required for installation

The following items must be prepared according to the “Installations Requirement Document”：

- Bench space and bench load capacity
- Environmental conditions
- Gas supplies
- Exhaust and ventilation
- Mains supply

Local work protection regulations must be followed and have priority over recommendations given in this manual.

■ Environmental Conditions

The Instruments AA-Spectrometer and its accessories are designed for indoor use and should be operated under follow condition:

• In a chemical laboratory with a minimum of vibrations /dust/corrosive vapors • Avoid direct sunlight

- Avoid placing instrument near strong electromagnetic fields
- At a room temperature of 10°C -35°C
- Within an atmosphere of a relative humidity between 20%–80% (non-condensing)

Statement:

Instrument company accepts no responsibility for damage or lack of performance due to poor operating conditions.

■ Space Requirements

The work bench must support at least 250Kg. Other optional accessories must be considered. There should be a gap of at least 40cm behind the instrument for ventilation and maintenance purposes.

➢ Dimensions And Weights

Instrument	Size			Mass
	Depth	Width	Height	
AAS	830	650	560	90Kg
CVG（opt）	-	-	-	-
PC	-			
Printer	-			

➢ Exhaust

Do not operate the AAS without an exhaust unit! Switch on the exhaust unit during operating furnace chemical vapor generator or hydride system!

AAS instruments need a ventilation system to the outside of the building, located above the atomizer compartment, in order to remove fumes emitted from flame, furnace or chemical vapor generator during operation. The exhaust hood should be 900mm above the bench and an exhaust fan capacity of at least 10m^3/min is needed. The fume hood and connecting tubing should be made of stainless steel.

➢ Power Requirement

The AAS operates from a single-phase AC mains power supply.

Voltage: 220volts-240 volts

Frequency: 50 Hz or 60 Hz

Power consumption: 7.0KVA Maximum (220V * 30A)

➢ Waste Liquid Disposal

A waste bottle comes with the instrument (auto-sampler).

➢ Gas Requirements

Caution!

Gases used in flame atomic absorption spectroscopy are ethyne, air and nitrous oxide. Use only instrument grade ethyne and nitrous oxide.

Caution!

Compressed gases should always be handled with care. follow the local safety regulations and manufacturer’s instructions provided by the supplier of the technical gases.

- Air

Air is used as the standard oxidant for AAS flame operation. Usually an air compressor is used to supply this standard oxidant. Air from pressurized cylinders can be used as well. Never use oxygen or oxygen-enriched air to operate your AAS!

- Ethyne

Ethyne is usually supplied in cylinders containing acetone. Only these pressurized gases must be used for operating Spectrum Instruments Flame AA Spectrometers. The pressure in the cylinder is not allowed to fall below 0.50 MPa (72.5 psi), as acetone may be carried into the gas box of the spectrometer. This may cause corrosion and malfunction of the system.

The rate of gas drawn from the cylinder should be moderate to prevent acetone from being carried with the ethyne. The ethyne used should at least be of “instrument grade” with a purity of 99.5%.

- Nitrous Oxide

Nitrous oxide is used as an oxidant, which increases the temperature of ethyne flames up to 2900°C.

Please read the instructions regard to the operation of N2O-C2H2 flames given in chapter one carefully before switching to this flame type.

The following specification lists the required gas supplies and gas connections:

Gas Type Flow	Flow Quality	Input Pressure	Rate (max.)
C2H2, Ethyne	2.5; 99.5%	0.1-0.12 MPa	300L/h
Air	Free of particles, oil, moisture	0.25-0.3 MPa	800L/h
N2O	2.5; 99.5 %	0.3-0.5 MPa	800L/h

Gas Type	Connector
C2H2	Connector for hose 9m
Air	Connector for hose 9mm or swagelock 6 mm
N2O	Connector for hose 9mm or swagelock 6 mm

- Installing The Nebulize

The nebulizer is packed separately for shipment and must be installed before using the burner system.

Before installing the nebulizer, make sure that the O-ring on the body is located properly and does not show any signs of damage.

It must be replaced immediately if damages, cracks or brittleness become visible.

- Installing The Tubing

Two capillaries are included in the nebulizer box. The capillary (inner diameter 0.7 mm) with one fixing screw and an open end is intended for manual flame operation. The capillary with two fixing screws (inner diameter 1.0 mm) is intended to be used with a flame auto- sampler. Connect the required capillary to the nebulizer.

The connection to the nebulizer gas (oxidant) is established via a cap-secured olive. Connect the oxidant tubing from the spectrometer with the nebulizer side arm and fix the screw.

- Connect the cylinder

Caution!

Gas supplies must be regulated with an appropriate reduction valve to maintain the instrument operating pressure. The gas must be dry and dust-free; otherwise a suitable filter should be inserted to the supply line.

- Argon

An inert gas is obligatory for operation of the graphite furnace. It is recommended to use only Argon as an inert gas connected to the 'Normal' gas inlet.

- Air

Air may be used at the alternate gas inlet. In GF-AAS it is usually supplied from a pressurized cylinder.

The following specification is listing the required gas supplies and gas connections.

Gas Type	Quality	Input pressure
Argon	99.999%	0.3±0.01MPa
Air	Free of particles, oil, moisture	0.2±0.01MPa

- Water

During installation of Furnace AAS, the service engineer will connect the unit to the cooling water supply. The pressure of the cooling unit should be between 0.09 MPa and 0.11 MPa (~27 psi to 30 psi).

A continuous supply of cooling water must be provided for furnace operation. If the furnace temperature exceeds the allowed maximum an interlock will stop the furnace heating. An error message and an acoustic signal will indicate the failure. The temperature must be below threshold before the furnace can be restarted again. If the error occurs repeatedly, check the graphite furnace program, as well as the temperature and the flow rate through the cooling system.

Note!

Depending on customer requirements at the time of installation, the alternate gas supply may not be connected. The alternate gas is connected to the respective gas inlet as required. Air or a mixture of 5% Hydrogen in Argon are the only gases recommended for operation with the Spectrum Instruments´ graphite furnace systems.

- Installation of The Graphite Tube

Graphite tubes are packed with every instrument. The tubes are free from contamination. Before using the furnace system, the tube has to be installed. Any contact of tubes shall wear gloves or use tools. Dedicated tools for graphite tube mounting are provided with the instrument. Always use this tool for installing and removing tubes from the furnace.

- **Lamps**

An individual Hollow Cathode Lamp (HCL) is required for each element determined. Spectrum Instruments offers Hollow Cathode Lamps for 68 elements with primary resonance lines between 193.7 nm and 780 nm.

Some elements can be combined in multi element Hollow Cathode Lamps. Please consult the list for accessories and consumables for availability. Prepare the lamps required for the analysis and install them into the lamp turret. For routine analysis the primary resonance wavelengths and the recommended lamp currents and slit widths are used.

They are default software settings.

- PC Requirements

Minimum Recommended PC configuration:

- CPU: PC with 2.66 GHz processor
- RAM: 4 GB Hard
- Drive: 500GB
- GPU: 256MB Graphics card supporting 1024 x 768 resolution
- DVD: 16 speed
- Audio: Integrated audio/sound card and speakers Keyboard and Mouse, 19-inch LCD Monitor Operation System: Windows 7 Professional 64-bit (Service Pack 1) operating system*
- RS-232 port for connection to the spectrometer

1.3 Software Installation

- Software Requirements

The software required to operate the Spectrum Instruments spectrometers and accessories and to handle the analytical data is the "RGWin-AA" file.

- Software Installation

- Insert the RGWin-AA CD into the CD-ROM drive
- Double click "my computer" and then you will find "RGWin-AA-setup"
- Double click it and you will find the setup program
- Double click on the setup Program (Setup.exe) and the software installation will commence

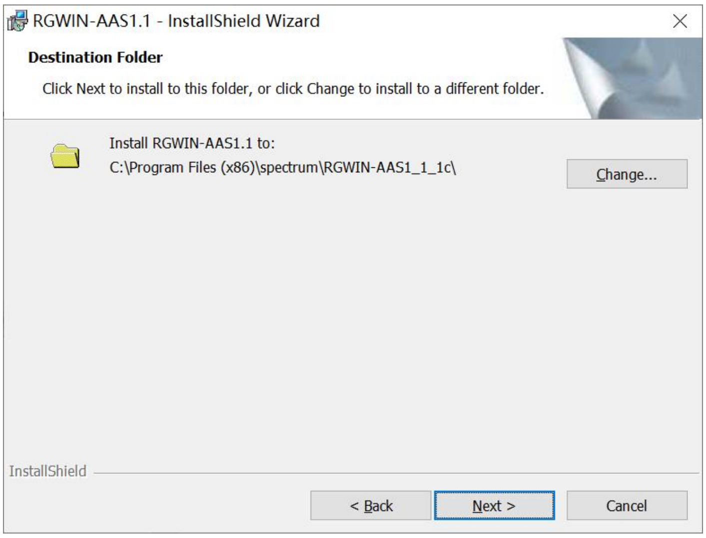

RGWIN-AAS1.1 - InstallShield Wizard

Welcome to the InstallShield Wizard for RGWIN-AAS1.1

The InstallShield(R) Wizard will install RGWIN-AAS1.1 on your computer. To continue, click Next.

WARNING: This program is for RG-AA Series only

- Select【Next】and then click on next;

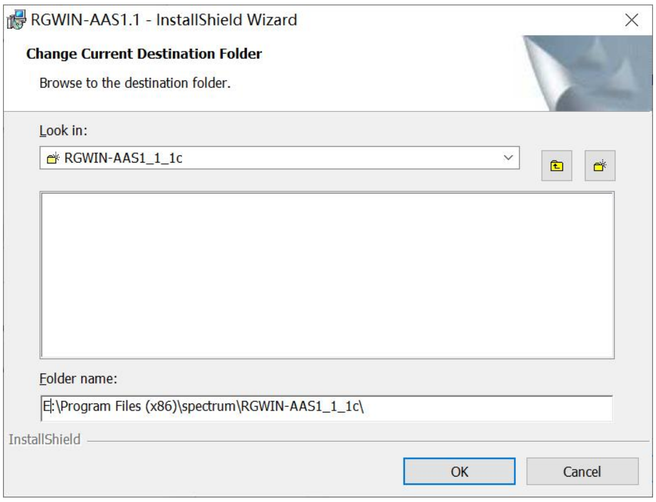

RGWIN-AAS1.1 - InstallShield Wizard

Destination Folder

Click Next to install to this folder, or click Change to install to a different folder.

Install RGWIN-AAS1.1 to:
C:\Program Files (x86)\spectrum\RGWIN-AAS1_1_1c\

- Select **Install dir** and press OK.



RGWIN-AAS1.1 - InstallShield Wizard

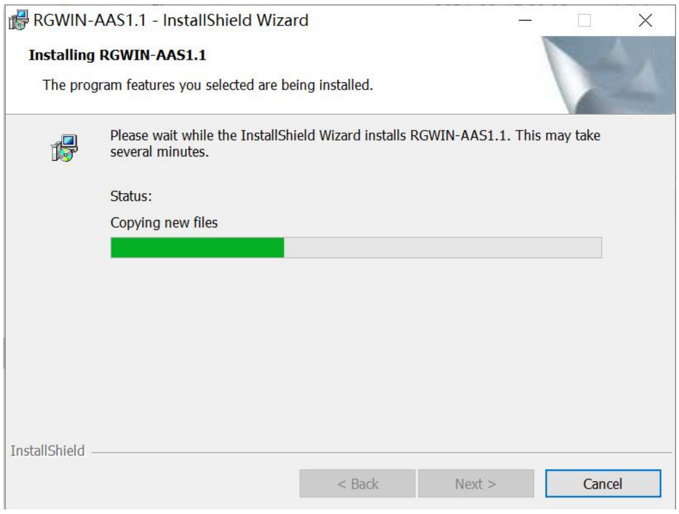
Change Current Destination Folder

Browse to the destination folder.

Look in: RGWIN-AAS1_1_1c

Folder name: E:\Program Files (x86)\spectrum\RGWIN-AAS1_1_1c\

- Select Next and press Next: copy file



RGWIN-AAS1.1 - InstallShield Wizard

Installing RGWIN-AAS1.1

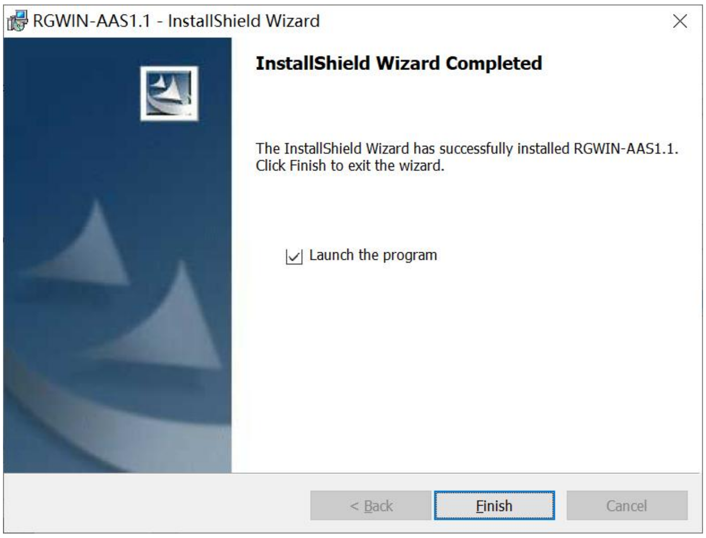
The program features you selected are being installed.

Please wait while the InstallShield Wizard installs RGWIN-AAS1.1. This may take several minutes.

Status:

Copying new files

- Finally select Finish and create shortcut on desktop.



RGWIN-AAS1.1 - InstallShield Wizard

InstallShield Wizard Completed

The InstallShield Wizard has successfully installed RGWIN-AAS1.1. Click Finish to exit the wizard.

☑ Launch the program

1.4 STARTING THE SPECTROMETER

■ Setup Checklist

To ensure the proper operation of the instrument, the system must first be set up correctly. Use the following checklist.

- Connect the components of the system with each other.
- Connect the equipment to the power supply.
- Install the hardware components as per your “analytical requirements” documents.

➢ Connections

A set of gas hose fittings and a mains cable which suits the common utility standard in your region is provided with your Spectrum Instruments AA- Spectrometer.

➢ Power

The mains power connection is located at the right side of the instrument. Refer to the next section for instructions on connecting the instrument to the mains power supply.

Before the instrument is connected to the power supply, ensure that the host and other accessories are powered off, and the air switch is also turned off.

After the instrument power cord is plugged into the power supply, turn on the air switch to the on state, and then turn on the main power supply of the instrument.

➢ Gas Hoses And Flame Gas Hoses

1) Flame Gas Hoses

Three gas hoses should be attached to the instrument via the fittings located on the left side of the instrument. Each fitting is color-coded for ethyne (red), air (black) and nitrous oxide (or auxiliary gas)(blue). The hoses should be firmly connected to the fittings and must be checked every time before starting the system. Replace the hoses when signs of aging detected.

2) Furnace Gas Hoses

Three gas hoses should be attached to the instrument via the fittings located at the left side of the instrument. Each fitting is color-coded for water (white), argon (gray) and air (black). The hoses should be secured on the fittings and must be checked every time before starting the system. Replace the hoses when signs of aging can be spotted.

➢ Accessories

For details on connection of accessories such as injection valve or auto-sampler, refer to the manuals shipped with the accessories.

- Starting System
- Pre-analysis Checklist

1. Install the required hardware, including optional accessories such as auto-sampler, according to the instruction in the related manuals for the accessories.

2. Turn on the instrument and possible peripheral accessories as described in the previous section.

3. Ensure that the venting system is working properly. To do this, hold a thin single-layer tissue close to the mouth of the extraction hood. The tissue should be drawn towards the hood.

4. Check the gas supplies to make sure you have enough gas to start the operation. Also ensure that the argon cylinder residual pressure is above 0.50 MPa (~72.5 psi).

5. Set the pressure at the relief valves to the following values

Flame

	Recommended		Permissible	
	kPa	Bar	kPa	Bar
Ethyne	100	1.0	100-130	1.0-1.3
Air	300	3.0	290-310	2.9-3.1
N2O	300	3.0	290-310	2.9-3.1

Furnace

	Recommended		Permissible	
	kPa	Bar	kPa	Bar
Ar	300	3.5	290-310	3.4-3.6
Air	300	3.0	290-310	2.9-3.1
Water	100	1.0	90-110	0.9-1.1

6. Check the gas hoses and replace any damaged hoses. Turn on the gas supplies and test all hoses and connections for leaks. Repair any leak immediately if found.

7. Start the software, create a new or stored method, optimize the system and start measurement. An introduction to the software can be found in the next chapter.

- Flame analysis Note:

If you use the air-C2H2 flame, a 100 mm or a 50 mm burner head can be used. If the N2O-C2H2 flame is required for the analysis, only the 50 mm burner head with scraper can be used. The flame type is selected in the software.

Chapter 2 RGWIN-AAS Software usage guide

2.1 Starting the RGWin-AA software

To start “RGWin-AA”software, click Start > Programs > RG > RGWIN-AAS 1.1

Alternatively, double-click on RGWIN-AAS 1.1 icon 。

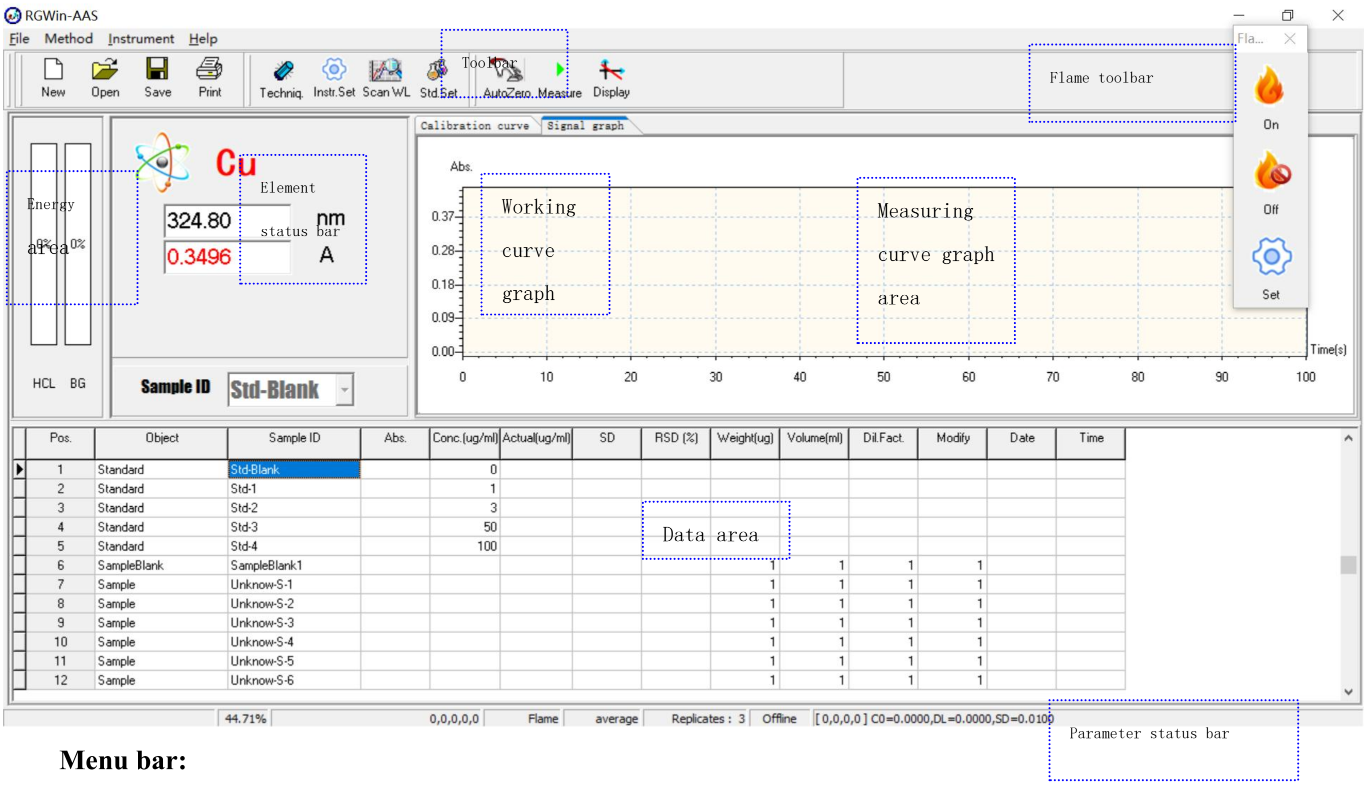
2.2 Interface and menu description

2.2.1 Program interface description

AA main program interface is the main operation interface you use. This interface provides you with all control options of "RGwin-aas" software. Through the simple, easy to operate and friendly operation interface, you can easily use "RGwin-aas" software without too much professional knowledge.

The main interface of the software consists of the following parts, mainly including:

- Menu bar
- Toolbar
- Information bar
- Calibration curve spectrum
- Real time absorption curve spectrum
- Work lamp and background correction energy display column
- Data result list display area and miscellaneous information column



Pos.	Object	Sample ID	Abs.	Conc.(ug/ml)	Actual(ug/ml)	SD	RSD (%)	Weight(ug)	Volume(ml)	Dil.Fact.	Modify	Date	Time
1	Standard	Std-Blank		0									
2	Standard	Std-1		1									
3	Standard	Std-2		3									
4	Standard	Std-3		50									
5	Standard	Std-4		100									
6	SampleBlank	SampleBlank1						1	1	1	1		
7	Sample	Unknow-S-1						1	1	1	1		
8	Sample	Unknow-S-2						1	1	1	1		
9	Sample	Unknow-S-3						1	1	1	1		
10	Sample	Unknow-S-4						1	1	1	1		
11	Sample	Unknow-S-5						1	1	1	1		
12	Sample	Unknow-S-6						1	1	1	1		

44.71% | 0,0,0,0,0 | Flame | average | Replicates : 3 | Offline | [0,0,0,0] C0=0.0000,DL=0.0000,SD=0.0100

Menu bar:

The window for menu operation includes four menu options [file], [method], [instrument] and [help]. Some information can be selected from this menu bar. The menu information is briefly introduced as follows:

File	This menu allows you to handle standard tasks such as opening, saving, exporting, and printing data. It also allows you to set the configuration of software related conditions, including printing settings, communication port settings, automatic sampler activation and graphic display settings.
Method	The method menu allows you to create / save / load methods and update data. You can also update the data and calibration curve after changing the correction method of the calibration curve. The measurement menu can change the relevant settings of test conditions and calculation methods.
Instrument	This menu contains the main working parameters of the instrument, such as the setting of working light, analysis wavelength, background correction method
Help	This menu can help, select language, and view the software version number

Toolbar:

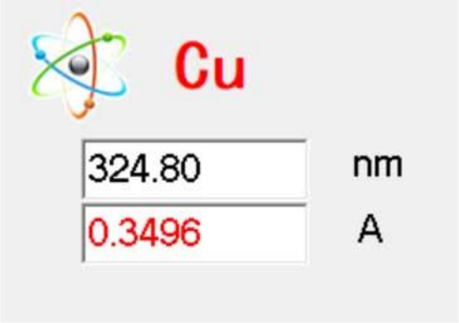
On the central operating page, provides tools and shortcuts to many common functions. The toolbar contains the buttons “new, open, save, print, technique, instrument settings, scan wavelength standard and sample settings, auto zero, measure and display”

The function of each button is as follows:

New	Create a new method
Open	Open a previously saved method
Save	Save the current method
Print	Print data
Technique	Choose technique
Instrument settings	Set the instrument parameters
Scan wavelength	Setting of wavelength and detector parameters
Standard and sample settings	Set parameters for standards and samples
Auto-zero	Set the actual absorbance value to zero
Measure	Start a determination
Display	Settings for the absorbance graphics

Element status bar:

The current sample number, element name, wavelength, sample absorbance (or absorbance after background correction) and background absorbance are displayed in this status bar.



Cu

324.80 nm

0.3496 A

Parameter status bar:

In this status bar, the corresponding values of current energy transmittance, current energy value, atomization mode, data calculation method, repeated measurement times, online status and mouse position in real-time graphics are displayed.

44.71%
0,0,0,0,0
Flame
average
Replicates : 3
Offline
[0,0,0,0] C0=0.0000,DL=0.0000,SD=0.0100

In this information column, the meanings of information from left to right are introduced as follows:

The first column shows the energy percentage of hollow cathode lamp after energy balance

The second column shows the relative counts of the measurement channel (hollow cathode lamp, any intensity unit) and the background channel (deuterium lamp, relative intensity unit or self absorption method, high-intensity pulse relative intensity unit). These data are the basis for calculating the energy intensity of the energy column.

The third column shows the analytical methods currently used (such as flame, graphite furnace and hydride generation)

The fourth column shows the data calculation method (peak height and peak area)

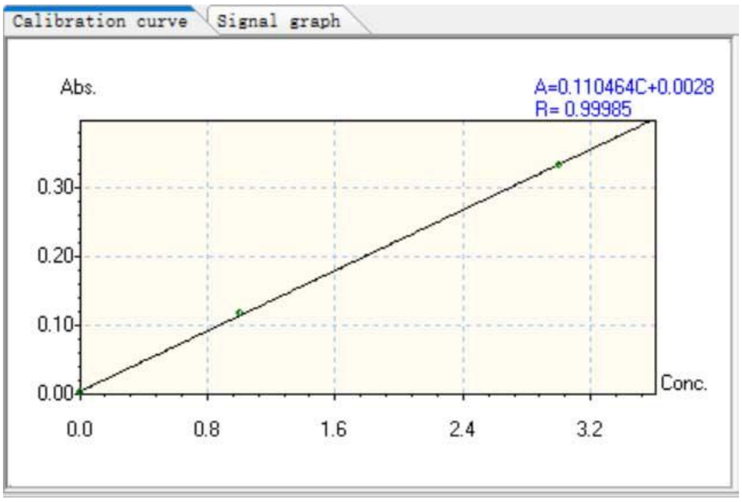
The fifth column shows the number of repetitions of the test

The sixth column shows the connection status between the instrument and the computer

The seventh column shows the data indicated by the cursor in the figure or data table.

Working curve graph area:

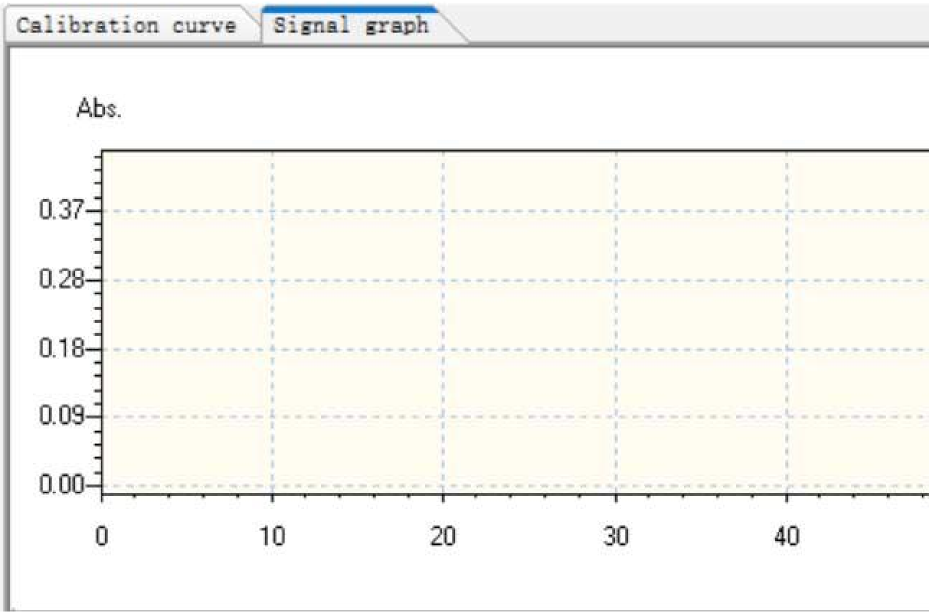
Display the working curve corresponding to the standard sample. When the standard blank and standard solution are measured, the software will automatically generate the calibration curve. The regression equation and correlation coefficient are shown in the calibration curve.



A=0.110464C+0.0028
R= 0.99985

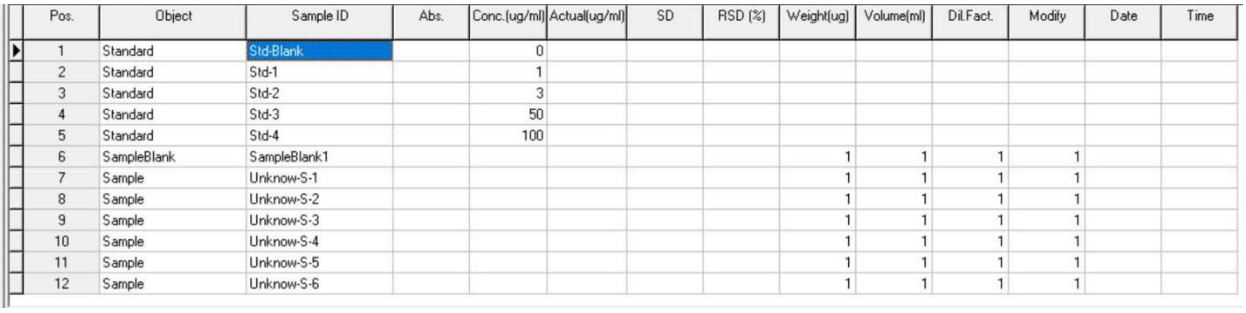
Measurement curve graph area:

Display the absorbance change curve during the sample test. In this chart, the current test signal will be displayed in real time.



Data area:

Absorbance values, results based on concentration units, and other related information is displayed in the data sheet. Additional information to further illustrate the type and preparation of the sample can be applied., the information includes the position of the samples, the sample names, the corrected absorbance as well as the background and the quantitative analytical results based on sample weight, dilution etc. Time and date of analysis are displayed.



Pos.	Object	Sample ID	Abs.	Conc.(ug/ml)	Actual(ug/ml)	SD	RSD (%)	Weight(ug)	Volume(ml)	Dil.Fact.	Modify	Date	Time
1	Standard	Std-Blank		0									
2	Standard	Std-1		1									
3	Standard	Std-2		3									
4	Standard	Std-3		50									
5	Standard	Std-4		100									
6	SampleBlank	SampleBlank1						1	1	1	1		
7	Sample	Unknow-S-1						1	1	1	1		
8	Sample	Unknow-S-2						1	1	1	1		
9	Sample	Unknow-S-3						1	1	1	1		
10	Sample	Unknow-S-4						1	1	1	1		
11	Sample	Unknow-S-5						1	1	1	1		
12	Sample	Unknow-S-6						1	1	1	1		

Pos.: the position of the standards and samples as defined in the auto-sampler

Object: reference solution, sample or blank

Sample ID: the name of the standards or samples

Abs.: the corrected absorbance (area or height)

BG: the background signal

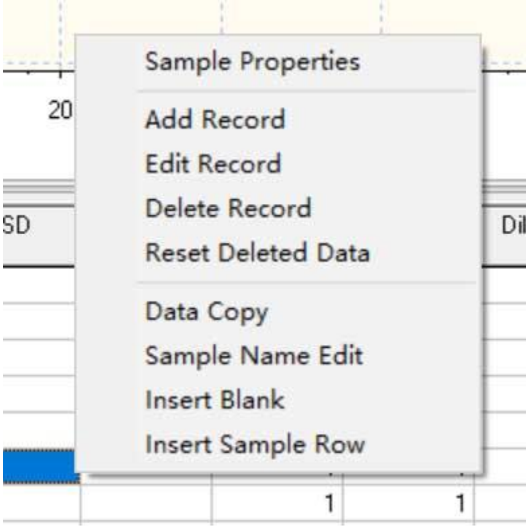
Conc.: the concentration of the sample

Actual: the recalculated concentration of the sample according to the weight, volume, and correction factors for e.g. dilution etc.

SD: Standard deviation

RSD: Relative standard deviation. Date/Time: the date/time of the measurement

Press right mouse in the data sheet. The following menu will be opened.

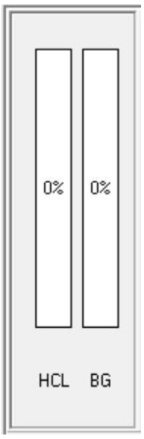


Sample properties:	The details of the result including the sample number, the concentration, wavelength, absorption, SD, RSD, number of replicates, corrected absorbance, total absorbance and background absorbance are displayed in this window.
Add record:	Add one more sample for determination
Edit record:	Input of the weight, volume, dilution times and factor of the sample
Delete record:	Delete the whole results of the sample
Reset deleted data:	Restore the deleted result
Data copy:	Copy the whole data in the data sheet
Sample name edit:	Edit the name of the sample
Insert Blank:	Insert the sample blank; the blank absorption will then be automatically deducted from the sample absorption
Insert sample row:	Insert an unknown sample

Intensity level bars for lamp and background corrector

Real-time intensity levels of the line-specific lamp and D2 lamp for background correction are displayed on this graphical display. In the case of Smith-Heftier background correction, the bars show matched intensities of the hollow-cathode lamps, both under normal and boost operating conditions. The bars should be matched close to 50%

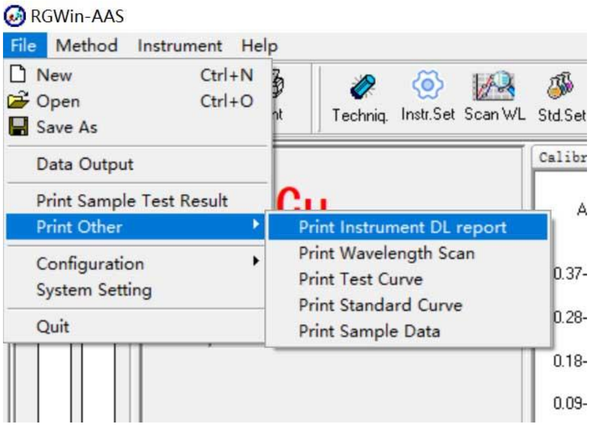
if no absorbance occurs. They serve as an indication of whether the intensities for background correction are matched properly by the instrument´s electronics. In the case of atomic or background absorbance the bars allow a rough estimate of the magnitude of absorbance. However, they are not intended for quantitative measurements.



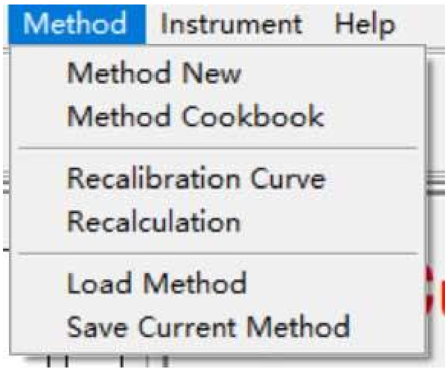
Flame toolbar:

Ignition, shutdown and setting. In the flame analysis mode window, you can click the "ignition" icon to ignite, and click the "turn off" icon to turn off the fire. Click the "setting" icon to open the window of gas flow rate, combustion head and other parameter settings, which can set the flow rate of gas and auxiliary gas, height and type of combustion head, scraper and other parameters.

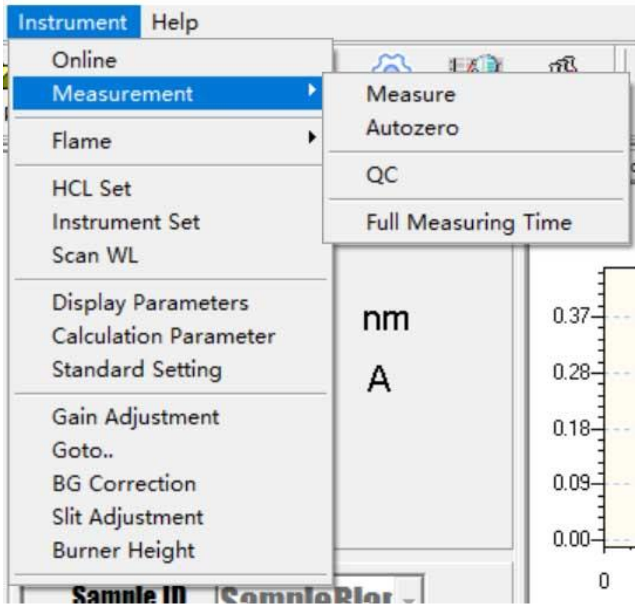
2.2.2 【File】Menu description



2.2.3 【Method】Menu description

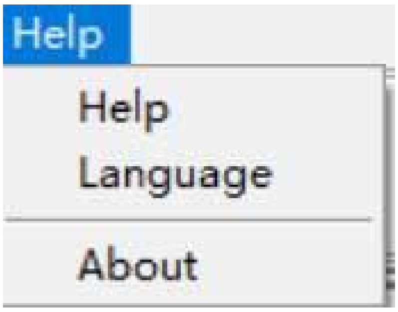


2.2.4 【Instrument】Menu description



2.2.5 【Help】Menu description

RGWIN-AAS The software provides detailed and thoughtful help functions (as shown in the figure). Through the [help] menu, you can open the [help topic] to get started quickly or solve difficult problems encountered during use; in the [language] menu, you can select Chinese and English; you can also select [about...] to view the version number, detailed address and contact information of the company.



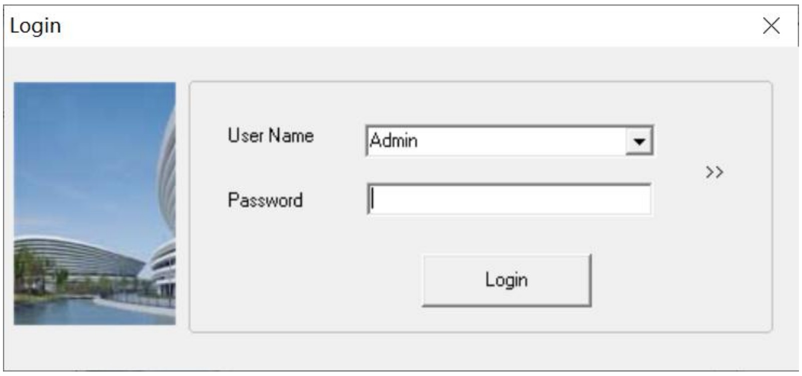
2.3 Operation

2.3.1 New working method

Based on the general usage of most ordinary users, the company has made reasonable default settings for RGwin-aas software. Therefore, ordinary users can measure elements without changing any other settings.

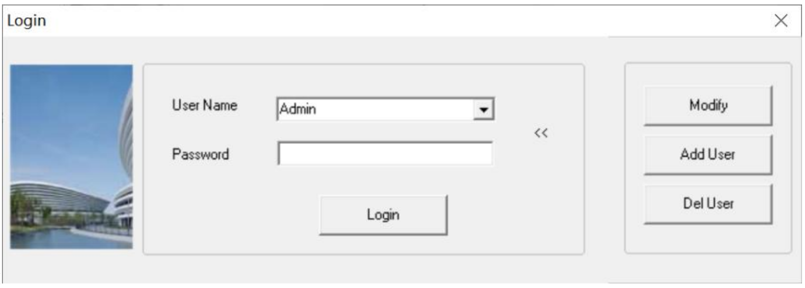
Step 1: Start the software

Step 2: User login: admin is the default user and the initial password is blank



User Name: Admin

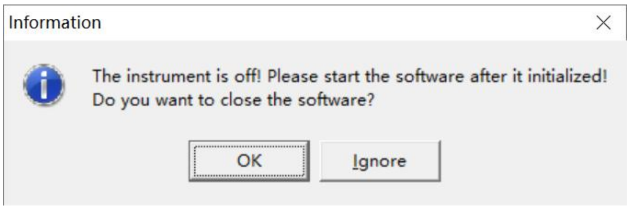
You can modify password, add user and delete user:



User Name: Admin

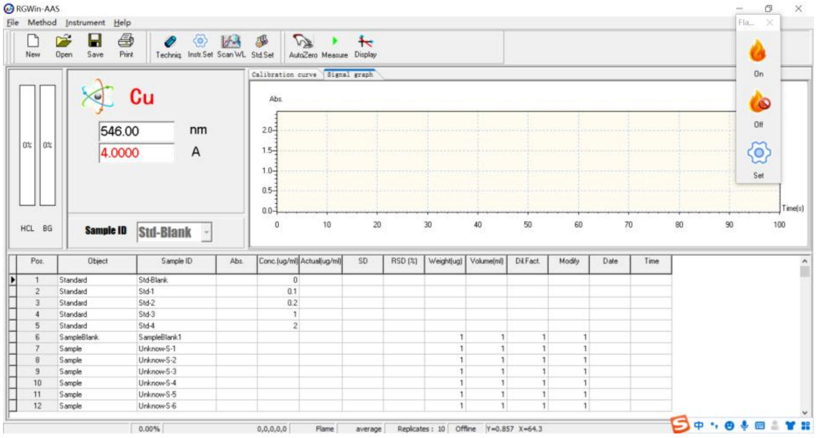
Step 3: Instrument model selection

Step 4: If you are not online, a prompt pops up. You can ignore [ignore]. If you are online, there is no prompt.



The instrument is off! Please start the software after it initialized!
Do you want to close the software?

Step 5: Enter the main interface

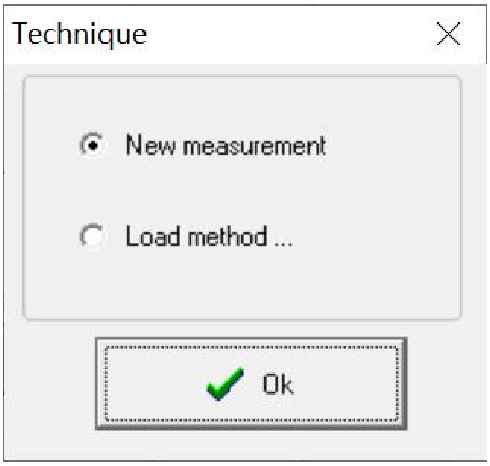


Cu
546.00 nm
4.0000 A
Sample ID Std-Blank

Pos.	Object	Sample ID	Abs.	Conc.(ug/ml)	Actual(ug/ml)	SD	RSD (%)	Weight(ug)	Volume(ml)	Dil.Fact.	Modify	Date	Time
1	Standard	Std-Blank		0									
2	Standard	Std-1		0.1									
3	Standard	Std-2		0.2									
4	Standard	Std-3		1									
5	Standard	Std-4		2									
6	SampleBlank	SampleBlank1						1	1	1	1		
7	Sample	Unknow-S-1						1	1	1	1		
8	Sample	Unknow-S-2						1	1	1	1		
9	Sample	Unknow-S-3						1	1	1	1		
10	Sample	Unknow-S-4						1	1	1	1		
11	Sample	Unknow-S-5						1	1	1	1		
12	Sample	Unknow-S-6						1	1	1	1		

0.00% 0,0,0,0,0 Flame average Replicates : 10 Offline Y=0.857 X=64.3

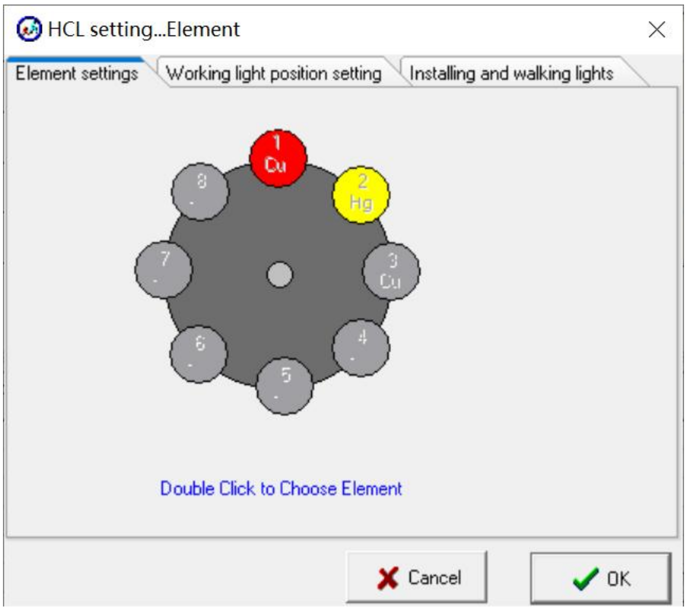
Step 6: Click “element” to create a new method



● New measurement

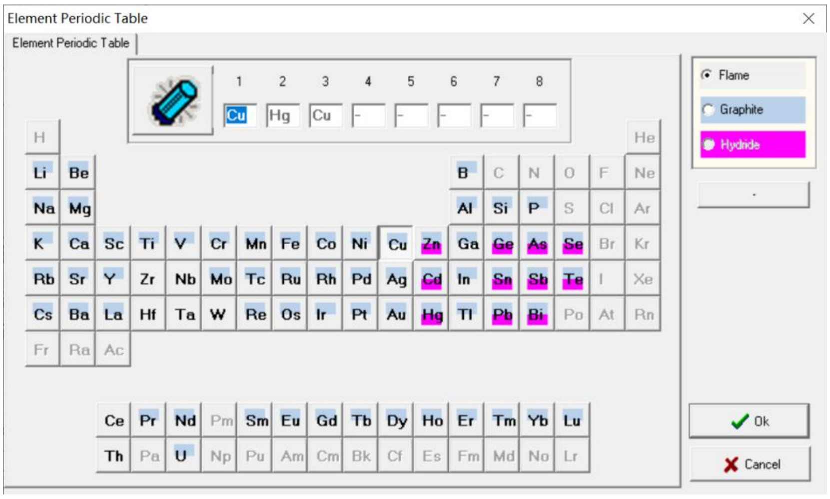
○ Load method ...

Step 7: Element setting, double-click the light bit to select the element



Double Click to Choose Element

Step 8: Select element, atomization mode



1	2	3	4	5	6	7	8
Cu	Hg	Cu	-	-	-	-	-

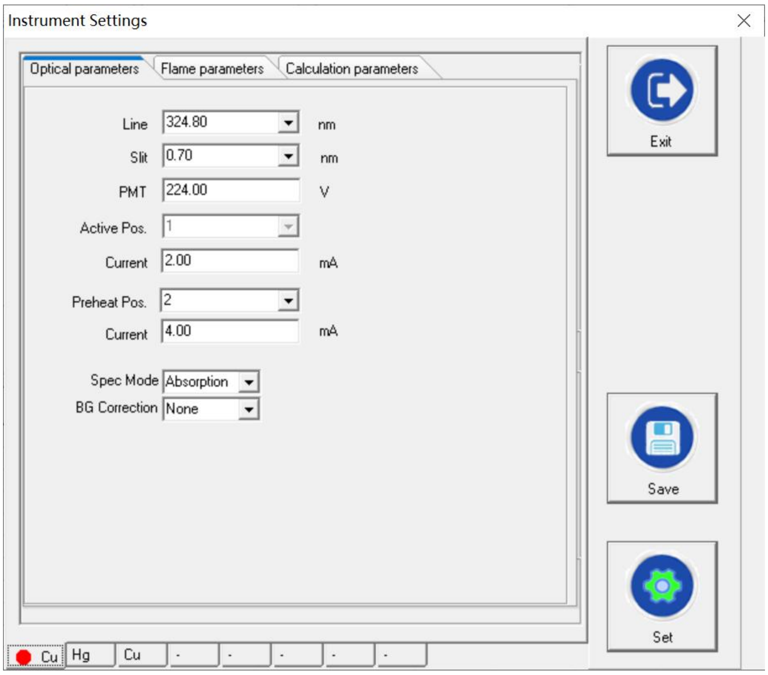
○ Flame

○ Graphite

● Hydride

Step 9: Click "Ok" to enter the "Condition Settings" window

Step 10: "Optical parameters" interface



Line 324.80 nm

Slit 0.70 nm

PMT 224.00 V

Active Pos. 1

Current 2.00 mA

Preheat Pos. 2

Current 4.00 mA

Spec Mode Absorption

BG Correction None

Optimization Of The Instrument Parameters:

Line: selection of the analytical line. The default setting is the most sensitive line of the element (primary resonance line). Others can be chosen from the drop box which are usually less sensitive that extend the working range of the instrument to higher concentrations. In rare cases, such as Pb which the default line is not the most sensitive, but it provides best signal to noise for complex samples.

Slit: choose the spectral bandwidth. Options are 0.1 nm, 0.2 nm,0.7 nm and 1.4 nm. The default value for the respective element will provide the best signal to noise ratio and the widest dynamic working range.

PMT: The PMT voltage of the photomultiplier tube, is set automatically. Changes to this setting is not recommended.

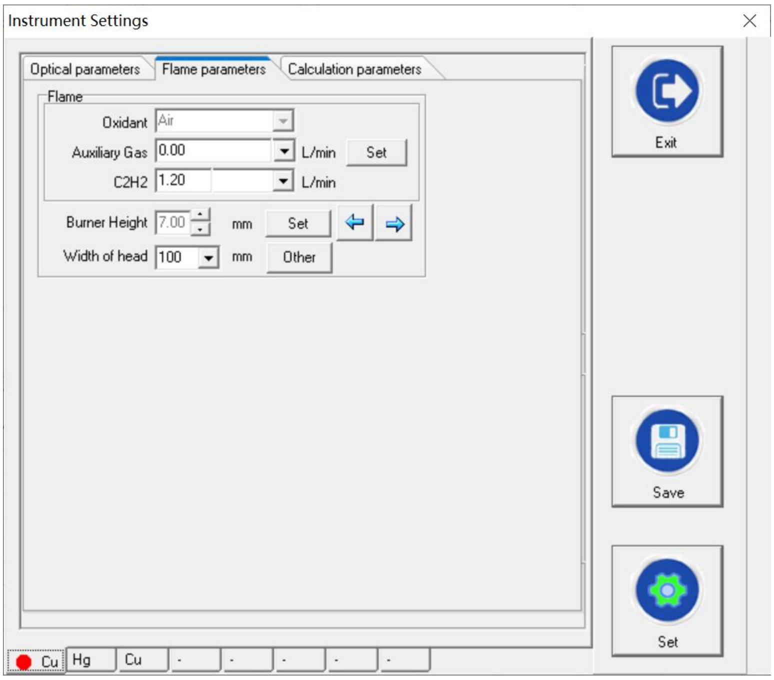
Active Pos.: The working lamp position is indicated here. The user may change the active position. Preheat pos.: Indication of the lamp position for pre-heating. Changes should be made in the lamp window only.

Meas. Mode: This window defines the mathematical operation with which the data from the detector are handled. “peak area” should be selected for Graphite furnace AAS measurements. In some cases “peak height” can be used as alternative in GF-AAS. The values recommended above are the default settings.

Replicates Mode: manual or auto. In “manual” operation the sample is injected by hand with a pipette. Auto means automatic injection with the sampler. Usually the auto sampler will be used for graphite furnace analysis.

Current: Allows to change the lamp current. It is strongly recommended to use the default parameters.
Background Correction: D2 lamp or S-H background correction can be selected
Spec.Mode: Absorption and Emission (AAS or AES) can be selected. Measurements in flame emission require adapted operation of the instrument.

Step 11: "Flame parameters" interface



Oxidant: Air
Auxiliary Gas: 0.00 L/min
C2H2: 1.20 L/min
Burner Height: 7.00 mm
Width of head: 100 mm

Oxidant:

Choose between Air or N2O

Oxidant Flow:

Flow rate of the additional oxidant gas. This function will increase the fixed oxidant flow through the nebulizer (~6L/min) by the value indicated in the drop box. The function is predominantly used, if energy-rich organic solvents, such as e.g. kerosene, are aspirated.

C2H2 Flow:

Flow rate of ethyne

Burner Height:

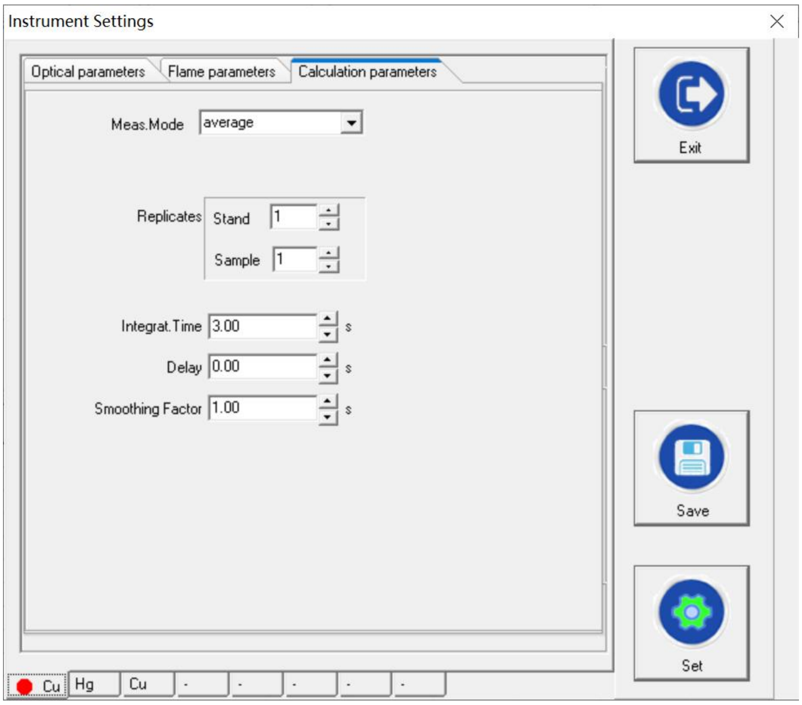
The distance between burner head and light beam can be adjusted from 5 mm to 25 mm.

Burner Type:

select the burner type, 50 mm or 100 mm. The 100 mm burner provides best sensitivity and detection limits for the air- ethyne flame. The 50 mm burner head can be used for air- ethyne operation as well. It will reduce sensitivity by about 1.7 x and thus extend the dynamic range to higher concentrations. Nitrous oxide- ethyne flames can only be operated with the 50 mm burner head.

Activate the “Other” button located on the right side to enable the scraper setting. (The scraper should always be used during the analysis using N2O-C2H2 flame). An interval of 60 s will be enough to run standard N2O-C2H2 flames with high long-term stability.

Step 12: “Calculation parameter” interface; After modifying various conditions, click the "setting" button to automatically set the wavelength, slit, lamp current and photomultiplier tube voltage, and enter the wavelength peak seeking interface;



Meas.Mode: average

Replicates: Stand 1; Sample 1

Integrat.Time 3.00 s

Delay 0.00 s

Smoothing Factor 1.00 s

Replicates Sample:

This selection defines the number of repetitive measurements for samples.

Replicates Std.:

This selection defines the number of repetitive measurements for blank and standards.

Integration Time:

The instruments is collecting a complete set of data each 20 ms. These are summed up for a certain amount of time which is defined with this function. A typical read time for a graphite furnace analysis is 3-5 s. The individual data are evaluated in various modes, are described above under the function “measurement mode”.

Delay:

Data collection is retarded by a certain amount of time in order to stabilize the analytical absorbance. In graphite furnace AAS only experienced users should apply this function.

Smoothing Factor:

no smoothing should be used in GFAAS. After all instrument settings are executed and automatic peak picking finishes, Close the scan WL window, and click to set standard and sample parameters.

Step 13: “Scan WL” interface;

Line 324.80 PMT 224.0

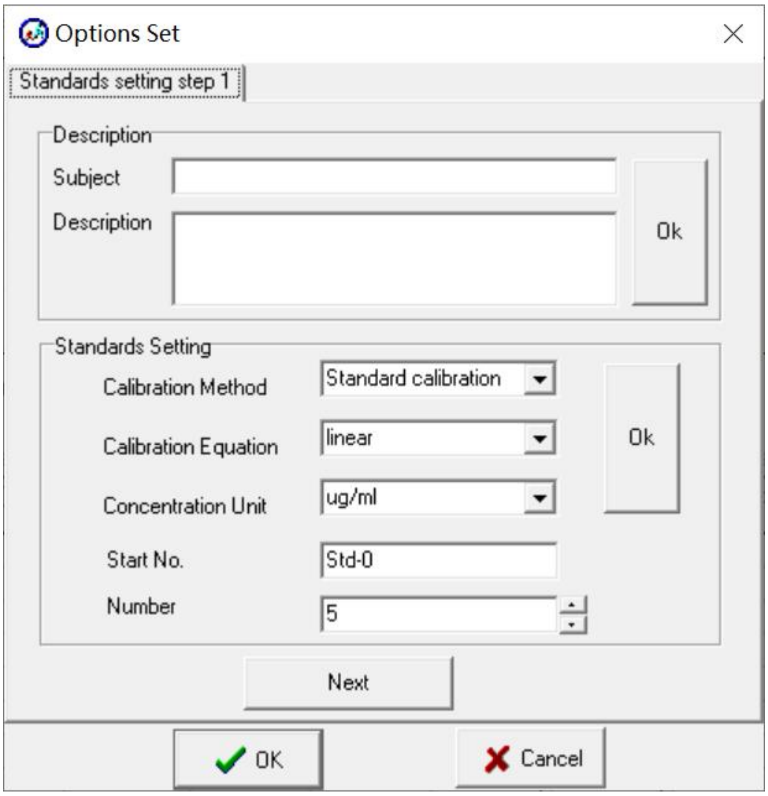
Current WL (nm) : 324.800

The instrument set-up routine will now automatically find the peak, set the optimal PMT voltage and gain, also balance the energies for measuring the total absorbance and background. Provided the standard D2 background correction, these are the intensities of HCL and D2 lamp. After the words “>>>>” disappear, the setting of the spectral parameters is finished. Click the “Close” icon to leave the window.

Step 14: If the wavelength peak is not ideal, you can click the "Scan start" button to automatically find the wavelength peak and find the wavelength position of the analysis line. After scanning, you can use the "Auto Gain" button to automatically adjust the high voltage to optimize the current energy；

Step 15: Start sample setup

Step 16: Click "standard sample" to enter the standard sample setting line window. Select the "correction method", "correction equation", "concentration unit", "standard sample start number", "standard sample quantity" and other settings of the calibration curve in turn. Press "next" to enter other settings



Calibration Method: Standard calibration
Calibration Equation: linear
Concentration Unit: ug/ml
Start No.: Std-0
Number: 5

Subject:

Alphanumerical field to describe the topic of the test.

Description:

Additional field for detailed information of the analysis.

Calibration Method:

Standard calibration method and Standard addition method. In most cases the standard calibration method will yield the most precise result with minimum time requirement. When the matrix in the sample is complex, high or is changing strongly from samples, it is difficult to prepare suitable matrix-matched standards. In this case, the standard addition method should be adopted. The standard addition method is used in situations where the sample matrix causes a multiplicative effect to the analytical signal. The standard addition method will not correct for additive effects, e.g. spectral interferences or blank levels.

Calibration Equation:

Equations based on a linear least squares algorithm or a nonlinear model can be selected. **Concentration unit:** select the concentration unit of the standards. The possible units are listed in the drop box.

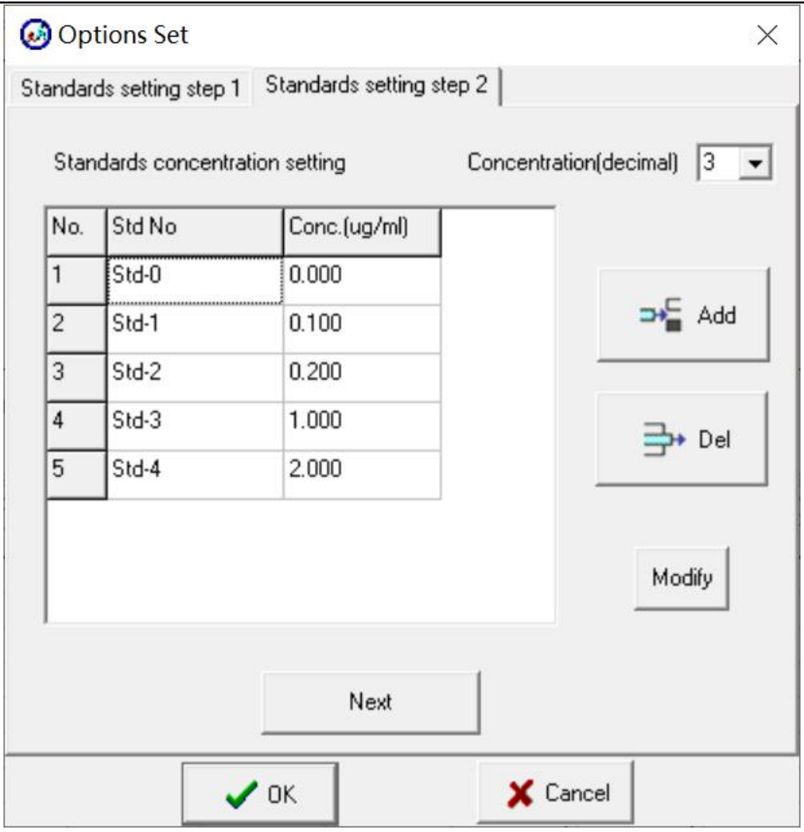
Number of Standards:

Up to 20 standard samples with different concentrations can be selected. The input must be in sequence from low to high concentration or vice versa.

Conc. Decimal:

The number of decimal digits is defined in this column. Up to 5 decimal points can be selected.

Step 17: Fill in the concentration of each standard point and press "next" to enter other settings



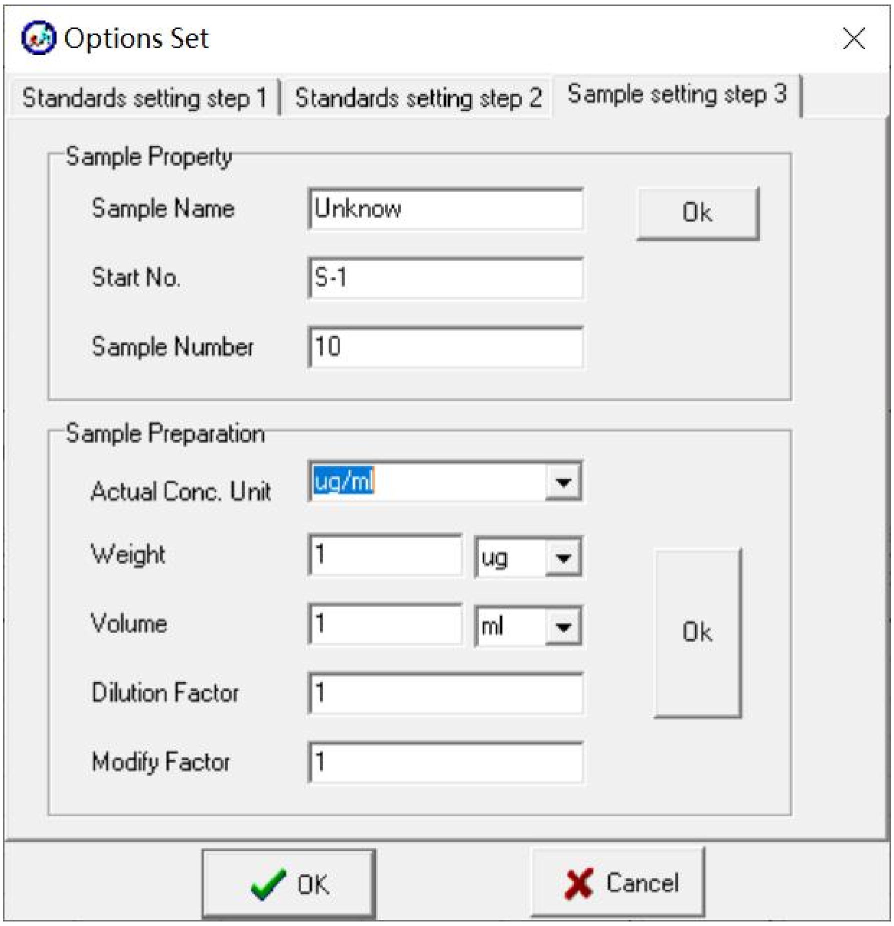
Standards concentration setting

Concentration(decimal) 3

No.	Std No	Conc.(ug/ml)
1	Std-0	0.000
2	Std-1	0.100
3	Std-2	0.200
4	Std-3	1.000
5	Std-4	2.000

Set the concentration for the standard solutions. Records can be added or deleted by clicking the Add or Del button. Click on Modify if you want to update the data sheet. After setting of the standards, page 2 will show up. Here is the description and the parameters for the samples.

Step 18: "sample setting": fill in the sample attribute and sample setting in turn; Press OK to submit and enter the main interface. At this time, the data in the table area will be refreshed to be empty.



Sample Property

Sample Name: Unknow

Start No.: S-1

Sample Number: 10

Sample Preparation

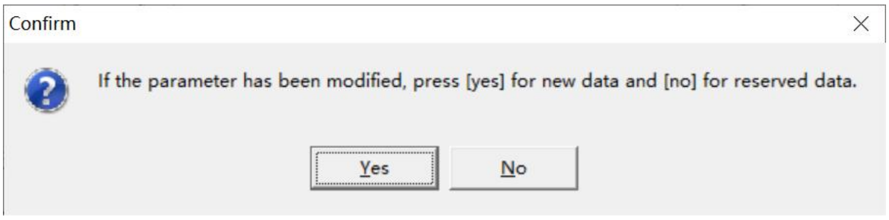
Actual Conc. Unit: ug/ml

Weight: 1 ug

Volume: 1 ml

Dilution Factor: 1

Modify Factor: 1



If the parameter has been modified, press [yes] for new data and [no] for reserved data.

Sample name: Sample name for the test

Start No.: The name and position of the first sample to be run

Sample number: The total number of samples to be analyzed.

Actual conc. unit: Concentration unit displayed on the screen and in the report

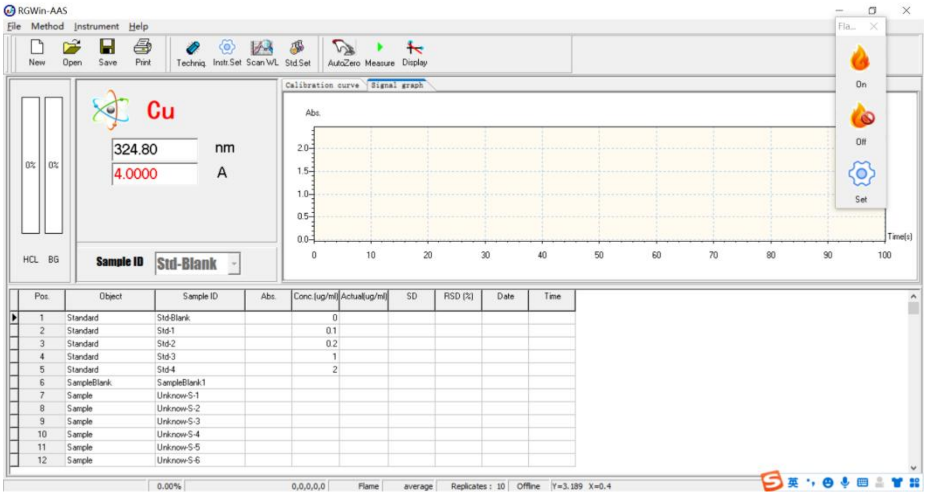
Weight: The weight of sample

Volume: The volume of sample

Dilution time: Dilution factor of the sample during the preparation.

Modify factor: Calibration factor. Calibrate test result and eliminated error.

Step 19: Create a new data table



324.80 nm

4.0000 A

Sample ID: Std-Blank

Pos.	Object	Sample ID	Abs.	Conc.(ug/ml)	Actual(ug/ml)	SD	RSD (%)	Date	Time
1	Standard	Std-Blank		0					
2	Standard	Std-1		0.1					
3	Standard	Std-2		0.2					
4	Standard	Std-3		1					
5	Standard	Std-4		2					
6	SampleBlank	SampleBlank1							
7	Sample	Unknow-S-1							
8	Sample	Unknow-S-2							
9	Sample	Unknow-S-3							
10	Sample	Unknow-S-4							
11	Sample	Unknow-S-5							
12	Sample	Unknow-S-6							

0.00% 0,0,0,0,0 Flame average Replicates : 10 Offline Y=3.189 X=0.4

Step 20: Start ignition:

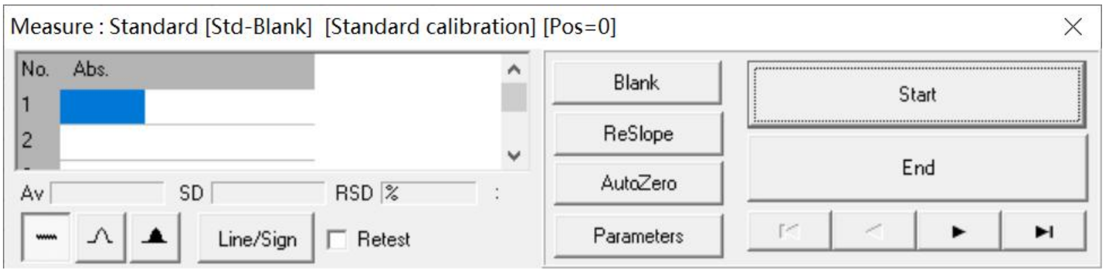
Open the air compressor and acetylene main valve, adjust the secondary air pressure to 2.5kg/cm2 (0.25MPa), adjust the secondary pressure of acetylene meter to 0.7kg/cm2 (0.07MPa), press the ignition switch in front of the combustion chamber or "On" in the computer software, and carefully adjust the acetylene flow after the flame is ignited to make the flame state meet the analysis requirements. If the flame absorbs large radiant energy from the light source, The "automatic energy regulation" operation can be carried out again.

Step 21: Auto Zero：Click AutoZero to Zero.

Step 22: Start The Measurement

Click “Measure” button from the Tool bar to start a measurement. The respective window “Measure” will open.

Press start, the auto sampler will run automatically. It will stop after an analysis cycle finished.



Measure : Standard [Std-Blank] [Standard calibration] [Pos=0]

Blank:

This function activates a blank reading; the system will stop after the first solution (the blank) has been determined. The blank value determined will be automatically subtracted from the samples measured later.

Start: Run a complete determination with blank, standards and samples as defined in the relevant page (see above).

Auto Zero:

sets the current absorbance to zero

ReSlope:

Recalculate the slope of the calibration curve by using a pre-defined standard solution. While the determinations are in progress, the “Measure page is filled with results:

Abs:

Numerical value of the corrected absorbance of the analyze- element after background correction. Depending on the selected measurement mode peak absorbance or integrated absorbance. Integrated absorbance is strongly recommended for graphite furnace determinations

Total:

the numerical value of the total absorbance determined by the hollow cathode lamp.

Step 23: Blank correction and slope re calibration can be carried out during sample measurement

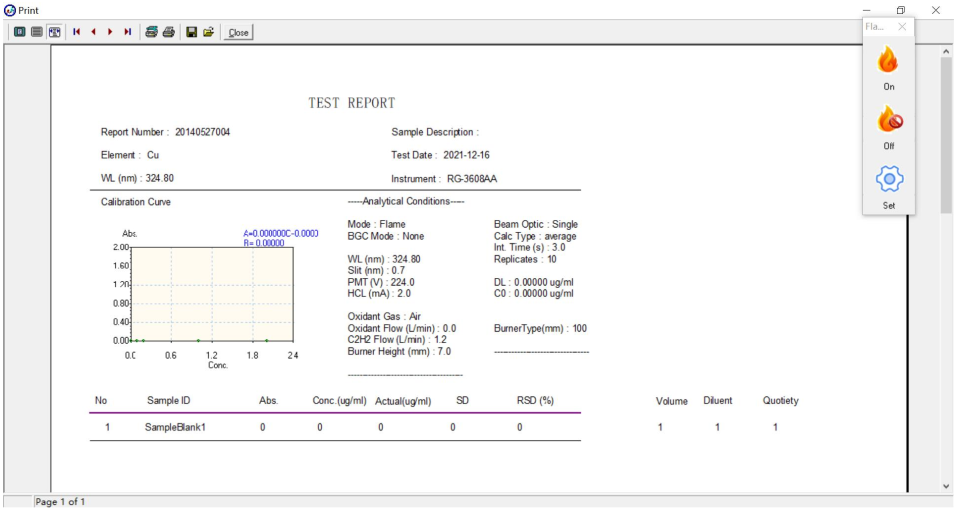
Step 24: After one element is measured, another element can be measured

Step 25: Click “End” to exit the measurement.

Step 26: Click Save to store data.

Step 27: After one element is measured, another element can be saved. To save this method, click method - > save current method in the main menu.

Step 28: Result printing, Click Print to enter print preview



TEST REPORT

Report Number : 20140527004
Sample Description :
Element : Cu
Test Date : 2021-12-16
WL (nm) : 324.80
Instrument : RG-3608AA

Calibration Curve

-----Analytical Conditions-----

Mode : Flame
BGC Mode : None
WL (nm) : 324.80
Slit (nm) : 0.7
PMT (V) : 224.0
HCL (mA) : 2.0
Oxidant Gas : Air
Oxidant Flow (L/min) : 0.0
C2H2 Flow (L/min) : 1.2
Burner Height (mm) : 7.0

Beam Optic : Single
Calc Type : average
Int. Time (s) : 3.0
Replicates : 10
DL : 0.00000 ug/ml
C0 : 0.00000 ug/ml
BurnerType(mm) : 100

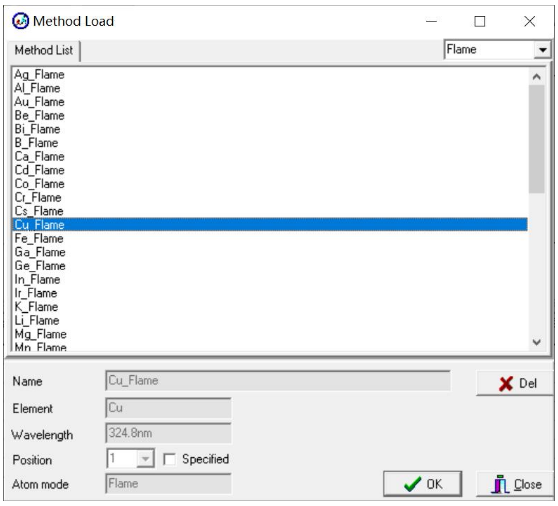
No	Sample ID	Abs.	Conc.(ug/ml)	Actual(ug/ml)	SD	RSD (%)	Volume	Diluent	Quotiety
1	SampleBlank1	0	0	0	0	0	1	1	1

Page 1 of 1

2.3.2 Measuring elements in "open working method" working mode

The commonly used measurement method of an element can be saved to the database. When it needs to be used, the method can be loaded for rapid measurement. When the method is loaded, the conditions of element determination, sample setting parameters and standard curve data are loaded at the same time.

Step 1：Load **method**：Main menu【**method**】|【Load **method**】

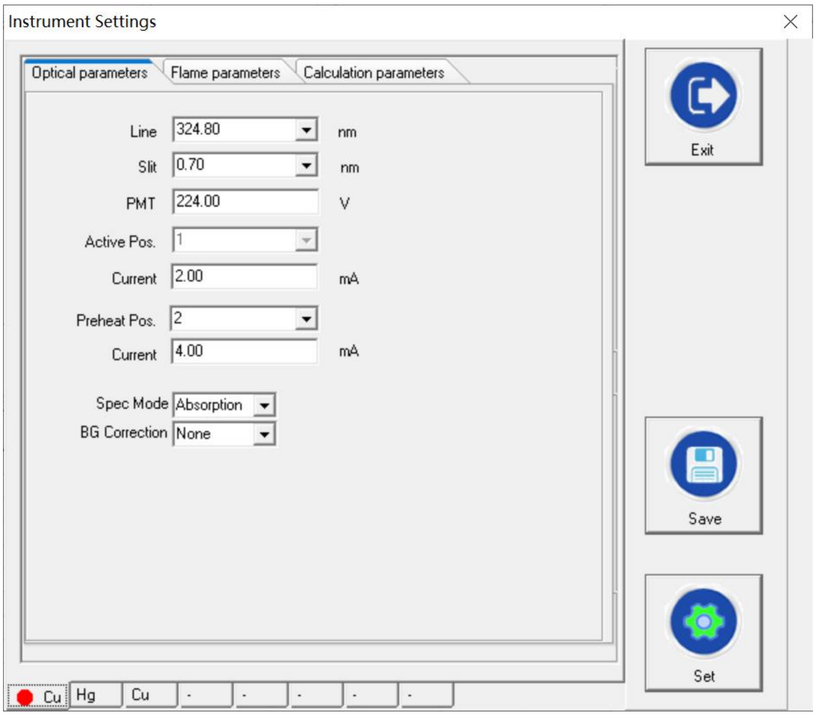


Flame

Ag_Flame
Al_Flame
Au_Flame
Be_Flame
Bi_Flame
B_Flame
Ca_Flame
Cd_Flame
Co_Flame
Cr_Flame
Cs_Flame
Cu_Flame
Fe_Flame
Ga_Flame
Ge_Flame
In_Flame
Ir_Flame
K_Flame
Li_Flame
Mg_Flame
Mn_Flame

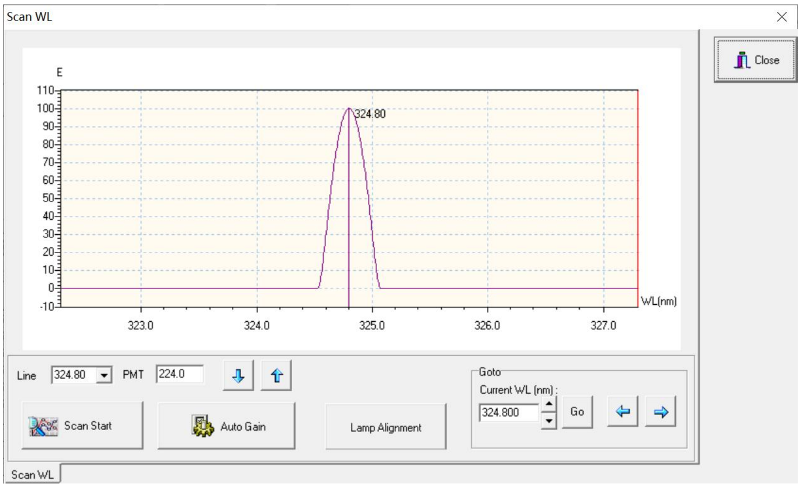
Name: Cu_Flame
Element: Cu
Wavelength: 324.8nm
Position: 1 ☐ Specified
Atom mode: Flame

Step 2："Condition Settings"



Line: 324.80 nm
Slit: 0.70 nm
PMT: 224.00 V
Active Pos.: 1
Current: 2.00 mA
Preheat Pos.: 2
Current: 4.00 mA
Spec Mode: Absorption
BG Correction: None

Step 3：“Scan WL”



Line 324.80

PMT 224.0

Current WL (nm) : 324.800

Step 4：Start ignition:

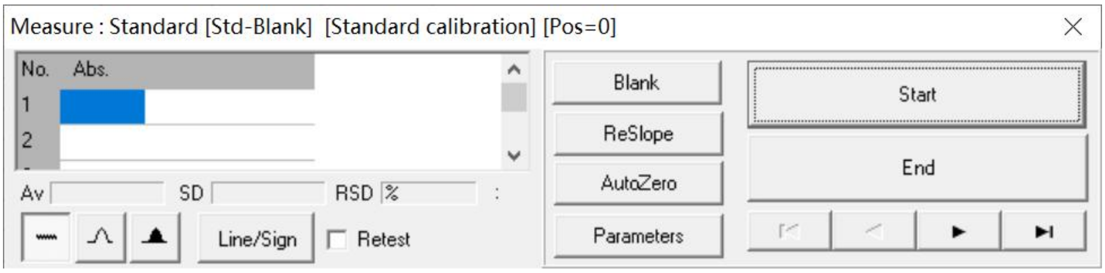
Open the air compressor and acetylene main valve, adjust the secondary air pressure to 2.5kg/cm2 (0.25MPa), adjust the secondary pressure of acetylene meter to 0.7kg/cm2 (0.07MPa), press the ignition switch in front of the combustion chamber or "On" in the computer software, and carefully adjust the acetylene flow after the flame is ignited to make the flame state meet the analysis requirements. If the flame absorbs large radiant energy from the light source, The "automatic energy regulation" operation can be carried out again.

Step 5: Auto Zero：Click AutoZero to Zero.

Step 6: Start The Measurement

Click the “Measure” button from the Tool bar to start a measurement. The respective window “Measure” will open.

Press start, the auto sampler will run automatically. It will stop after an analysis cycle finished.



Step 7: Blank correction and slope re calibration can be carried out during sample measurement

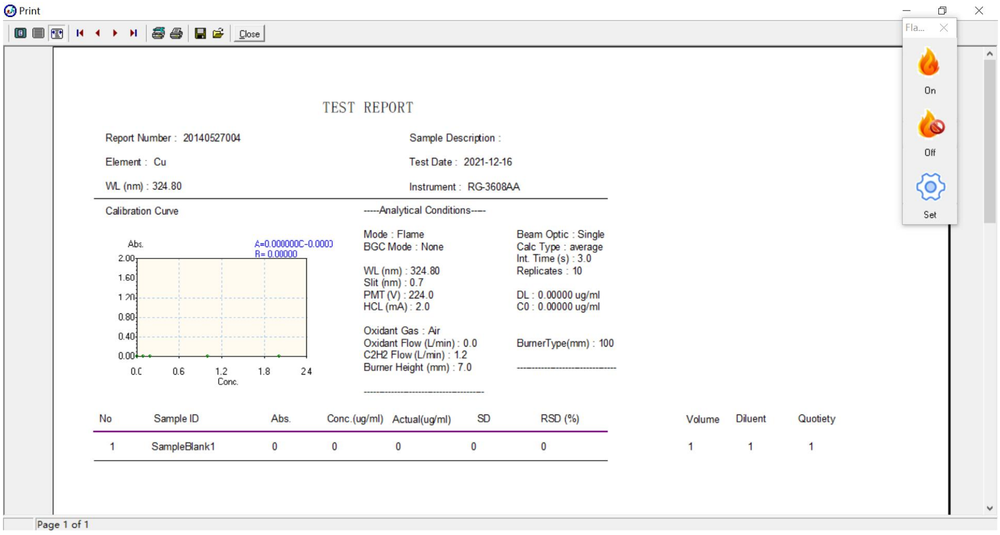
Step 8: After one element is measured, another element can be measured

Step 9: Click “End” Exit the measurement.

Step 10: Click Save to store data.

Step 11: After one element is measured, another element can be saved. To save this method, click method - > save current method in the main menu.

Step 12: Result printing，Click Print to enter Print preview



TEST REPORT

Report Number : 20140527004
Sample Description :
Element : Cu
Test Date : 2021-12-16
WL (nm) : 324.80
Instrument : RG-3608AA

Calibration Curve

-----Analytical Conditions-----

Mode : Flame
BGC Mode : None

WL (nm) : 324.80
Slit (nm) : 0.7
PMT (V) : 224.0
HCL (mA) : 2.0

Oxidant Gas : Air
Oxidant Flow (L/min) : 0.0
C2H2 Flow (L/min) : 1.2
Burner Height (mm) : 7.0

Beam Optic : Single
Calc Type : average
Int. Time (s) : 3.0
Replicates : 10

DL : 0.00000 ug/ml
C0 : 0.00000 ug/ml

BurnerType(mm) : 100

No	Sample ID	Abs.	Conc.(ug/ml)	Actual(ug/ml)	SD	RSD (%)	Volume	Diluent	Quotiety
1	SampleBlank1	0	0	0	0	0	1	1	1

2.4 Introduction RGwin-AAS software function window

2.4.1 Energy adjustment window: main menu [instrument] [energy adjustment]

2.4.1.1 working lamp current: the current of the hollow catenary lamp in the No. 1 lamp position.

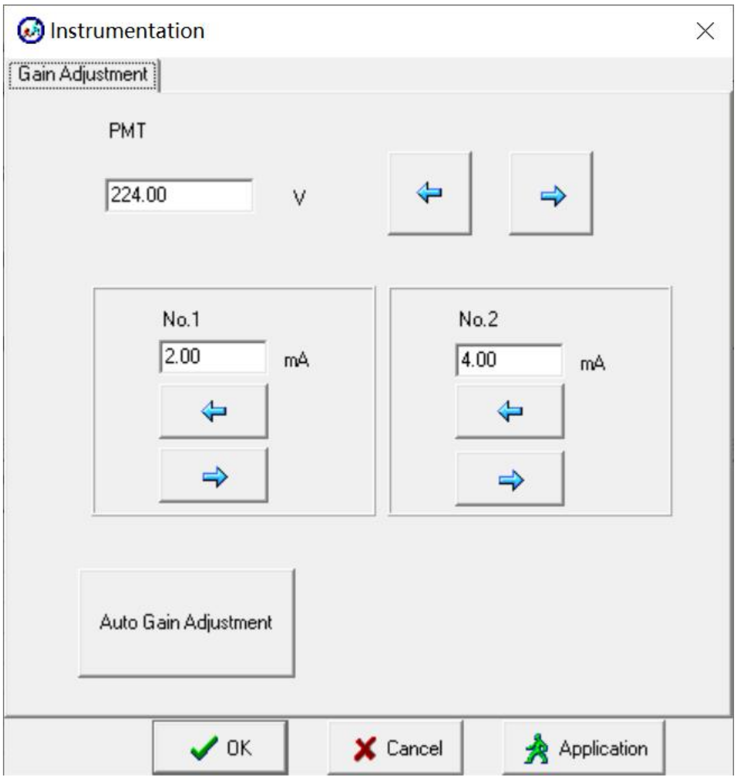
2.4.1.2 Preheat lamp current: the current of the hollow catenary lamp in the No. 2 lamp position.

2.4.1.3 High voltage: the working (negative) high pressure of the photomultiplier tube.

2.4.1.4 Background balance: When the background is corrected, if the energy indication of the deuterium lamp or the hollow yin lamp is too different, you can press this key to balance the energy of the two beams of light.

2.4.1.5 Apply key: change settings without exiting the window

2.4.1.6 Confirm key: Exit the window and change the settings



PMT 224.00 V

No.1 2.00 mA

No.2 4.00 mA

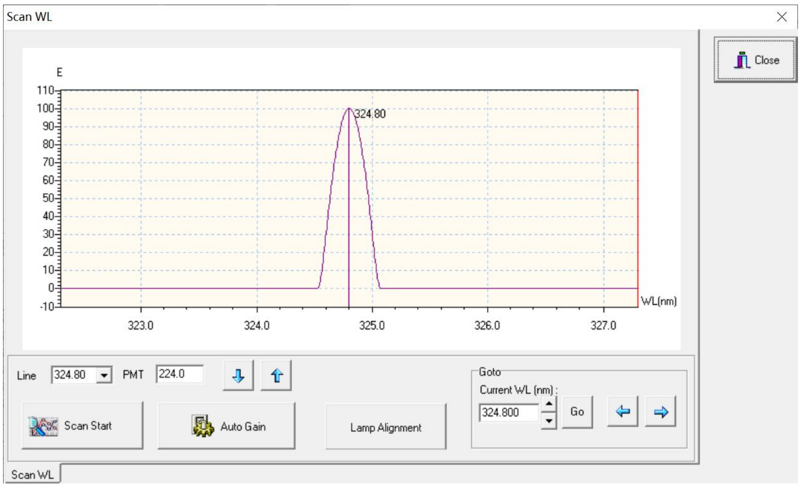
2.4.2 Wavelength Peaking Window: Main Menu [Instrument] [Auto Peaking]

2.4.2.1 Start Scanning: Use the wavelength value and high voltage value in the current window for wavelength scanning, and automatic peaking.

2.4.2.2 Auto Adjust Energy: This key automatically adjusts the energy to an appropriate value.

2.4.2.3 Go: Moving wavelength positions.

2.4.2.4 Automatic trimming of lamp position: The lamp position is automatically fine-tuned to the optimal position.



Line: 324.80

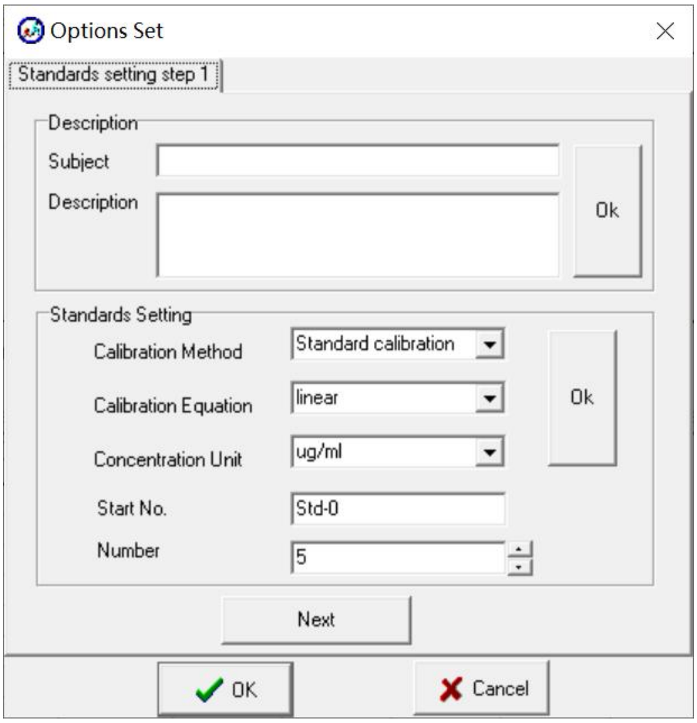
PMT: 224.0

Current WL (nm): 324.800

2.4.3 Sample Setup Window

2.4.3.1 Go to: Main Menu [Measurement] | [Standard Sample Settings] or Toolbar, click Std. Set.

2.4.3.2 Standard Sample Setup 1 Content



Calibration Method: Standard calibration

Calibration Equation: linear

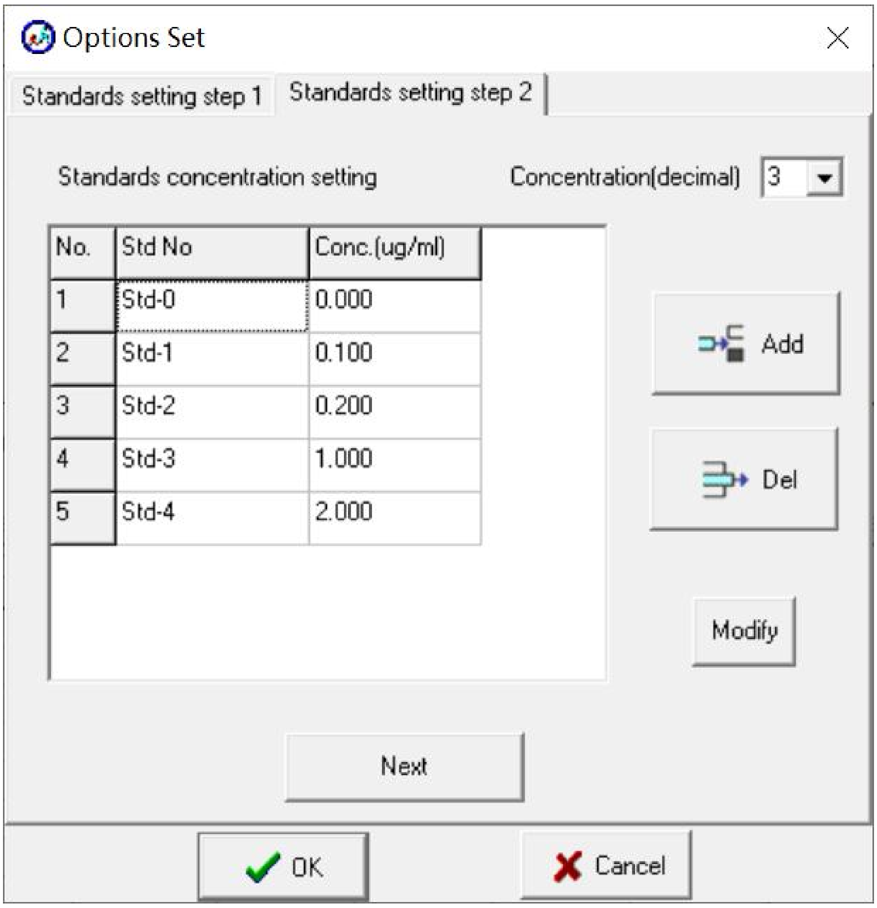
Concentration Unit: ug/ml

Start No.: Std-0

Number: 5

- **Subject:**
- Alphanumerical field to describe the topic of the test.
- **Description:**
- Additional field for detailed information of the analysis.
- **Calibration Method:**
- Standard calibration method and Standard addition method. In most cases the standard calibration method will yield the most precise result with minimum time requirement. When the matrix in the sample is complex, high or is changing strongly from samples, it is difficult to prepare suitable matrix-matched standards. In this case, the standard addition method should be adopted. The standard addition method is used in situations where the sample matrix causes a multiplicative effect to the analytical signal. The standard addition method will not correct for additive effects, e.g. spectral interferences or blank levels.
-
- **Calibration Equation:**
- Equations based on a linear least squares algorithm or a nonlinear model can be selected. **Concentration unit:**
- select the concentration unit of the standards. The possible units are listed in the drop box.
- **Number Of Standards:**
- Up to 20 standard samples with different concentrations can be selected. The input must be in sequence from low to high concentration or vice versa.
- **Conc. Decimal:**
- The number of decimal digits is defined in this column. Up to 5 decimal points can be selected.

2.4.3.3 Standard sample setup 2 contents.



Concentration(decimal) 3

No.	Std No	Conc.(ug/ml)
1	Std-0	0.000
2	Std-1	0.100
3	Std-2	0.200
4	Std-3	1.000
5	Std-4	2.000

Set the standard solution concentration. You can click the Add Line, Delete Row icon to add or delete records. Click the "Modify" icon to update the concentration setting.

2.4.3.4 Sample set 3 contents

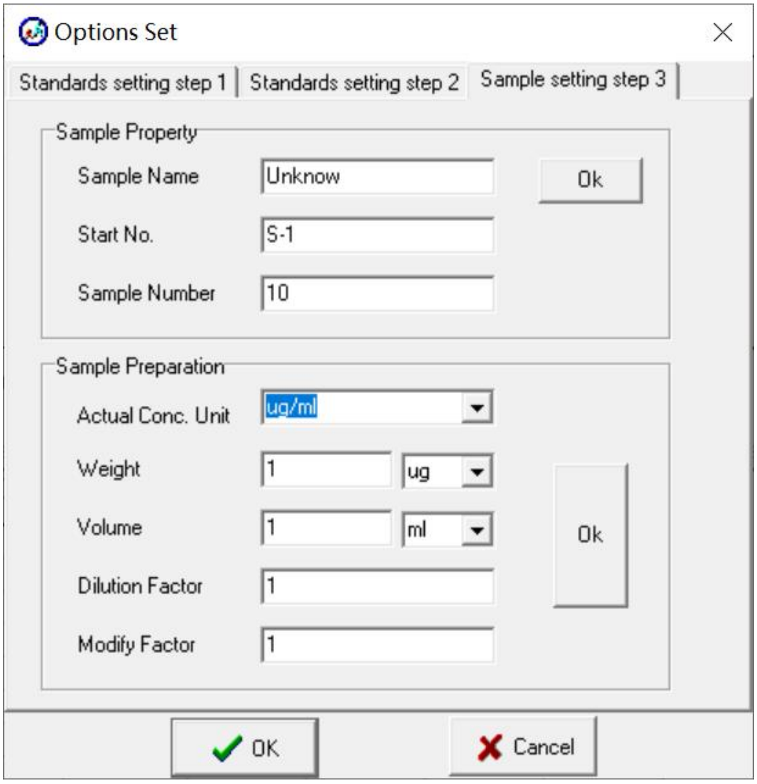
On this page, you can set the name, quantity, sample start number, and actual concentration units, and the coefficients necessary to calculate the actual concentration. In the measurement table, one of the items is "Actual Concentration", which is calculated as:

Actual concentration = concentration * (volume * dilution multiple * correction factor) / weight

Note: Volume here refers to the coefficient corresponding to the volume change when converting the actual concentration, and weight refers to the coefficient corresponding to the weight change when converting the actual concentration.

You can enter the corresponding coefficient according to the specific situation of the sample and calculate the actual concentration. When you open the Sample Setup Wizard, the default values for these factors are all 1. That is, when you do not make a setup, the actual concentration is equal to the concentration value after the measurement is over.

2.4.3.5 Modify the above coefficients: In the "Sample Settings" window, modify the coefficients, and then "Modify"; Remember, press the cancel key to exit, otherwise the data will be emptied。



Sample Name: Unknow
Start No.: S-1
Sample Number: 10
Actual Conc. Unit: ug/ml
Weight: 1 ug
Volume: 1 ml
Dilution Factor: 1
Modify Factor: 1

Sample name: Sample name for the test
Start No.: The name and position of the first sample to be run
Sample number: The total number of samples to be analyzed.
Actual conc. unit: Concentration unit displayed on the screen and in the report
Weight: The weight of sample
Volume: The volume of sample
Dilution time: Dilution factor of the sample during the preparation.
Modify factor: Calibration factor. Calibrate test result and eliminated error.

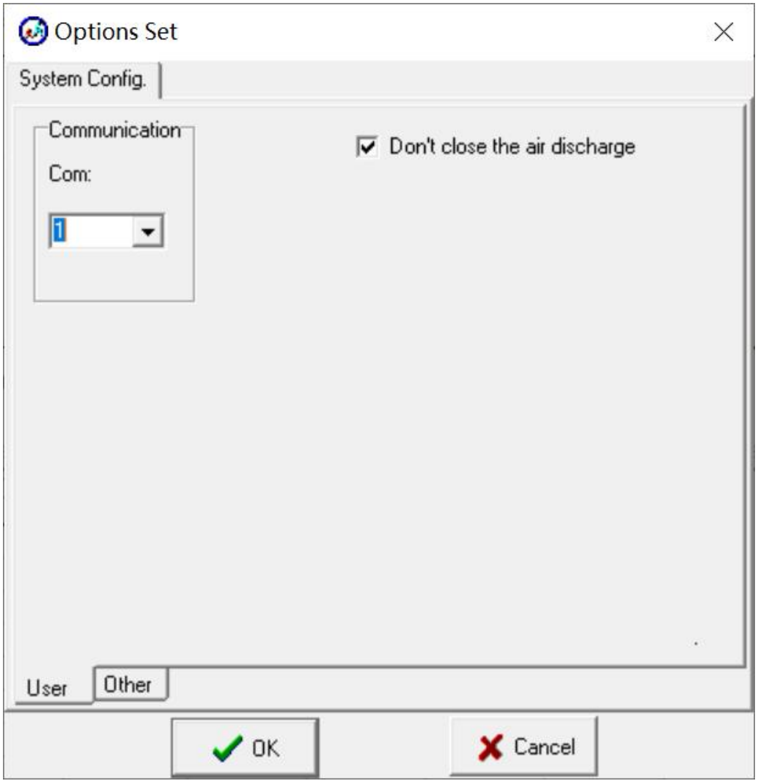
2.4.4 System Settings Window

2.4.4.1 System Settings Content

1.Commication port

2.Other Settings

2.4.4.2 Go: Main Menu [File] | [System Settings].



Com: 1

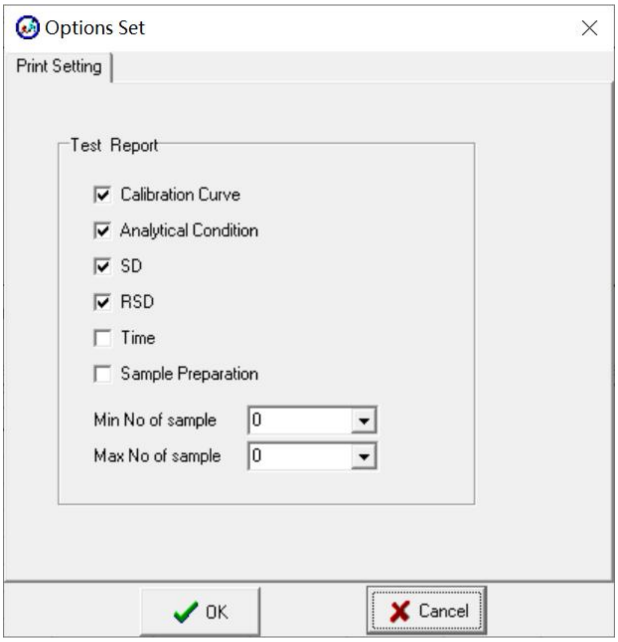
☑ Don't close the air discharge

2.4.5 “Print Set” window

2.4.5.1 “Print Set” Content

1. Work curve displayed in the results table
2. Test conditions displayed in the results
3. table shows individual sample configuration data
4. SD, RSD, time

2.4.5.2 Enter: The main menu [File] | [Option Settings] | [Print Settings].



☑ Calibration Curve
☑ Analytical Condition
☑ SD
☑ RSD
☐ Time
☐ Sample Preparation

Min No of sample: 0

Max No of sample: 0

2.4.6 Project Settings

2.4.6.1 Project Settings Content

1 Project name

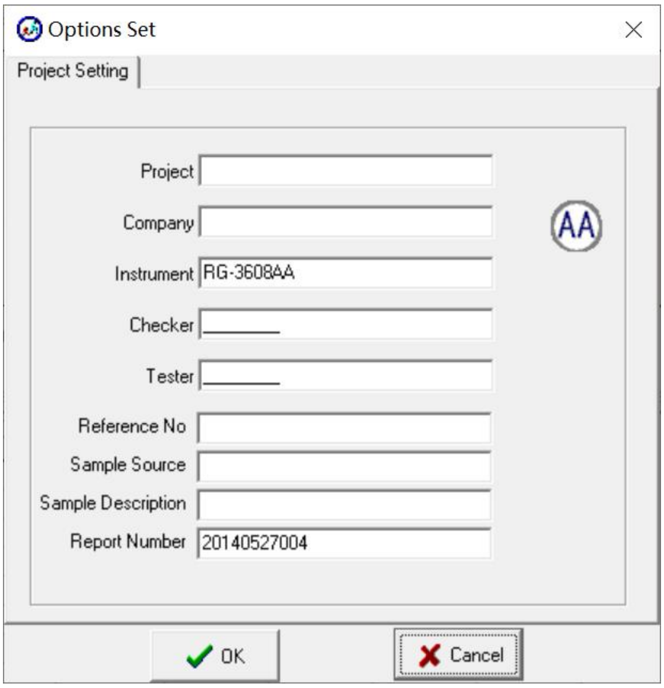
2 Unit name

3 Instrument name

4 Checker

5 Inspector

2.4.6.2Instruction: Main menu [File] | [Option Settings] | [Project Settings]



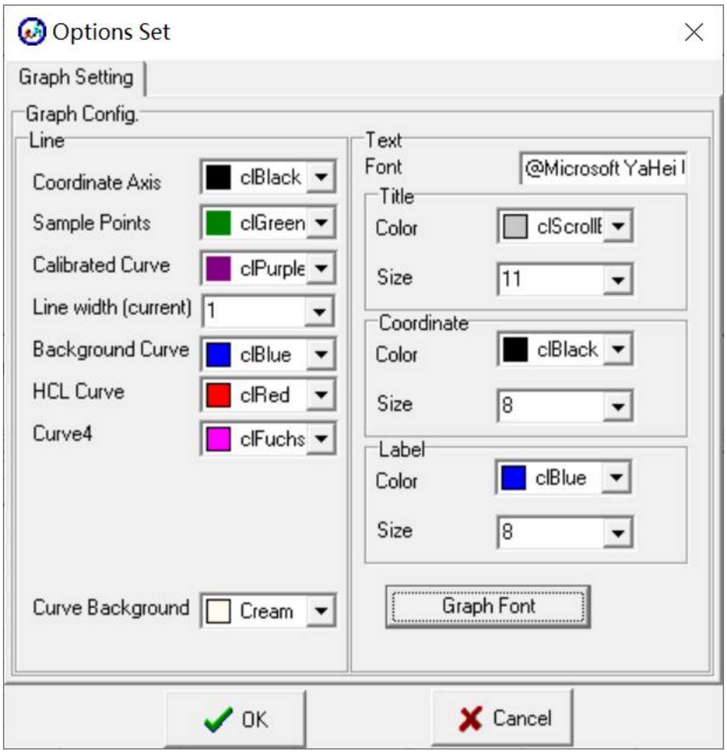
Project
Company
Instrument RG-3608AA
Checker ________
Tester ________
Reference No
Sample Source
Sample Description
Report Number 20140527004

2.4.7 Graphics Settings

2.4.7.1 Graphics Settings Content

1. Lines in the drawing: line width, color settings of coordinate axis, sample point and curve
2. Background in drawings: drawing background color settings
3. Text in graphics: font name, color and size settings of title, coordinates and dimensions

2.4.7.2Instruction: Main menu [File] | [Option Settings] | [Graphics Settings]



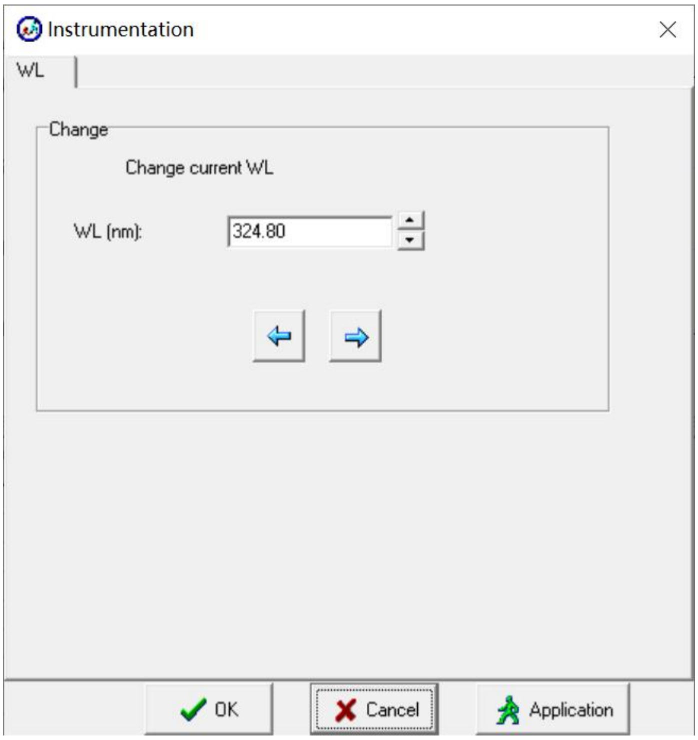
Coordinate Axis: clBlack
Sample Points: clGreen
Calibrated Curve: clPurple
Line width (current): 1
Background Curve: clBlue
HCL Curve: clRed
Curve4: clFuchs
Curve Background: Cream

Font: @Microsoft YaHei I
Title Color: clScrollE
Title Size: 11
Coordinate Color: clBlack
Coordinate Size: 8
Label Color: clBlue
Label Size: 8

2.4.8 Go Wavelength

2.4.8.1 Content: Changes the current wavelength position

2.4.8.2 Enter: Main menu [Instrument] | [Go Wavelength].



WL (nm): 324.80

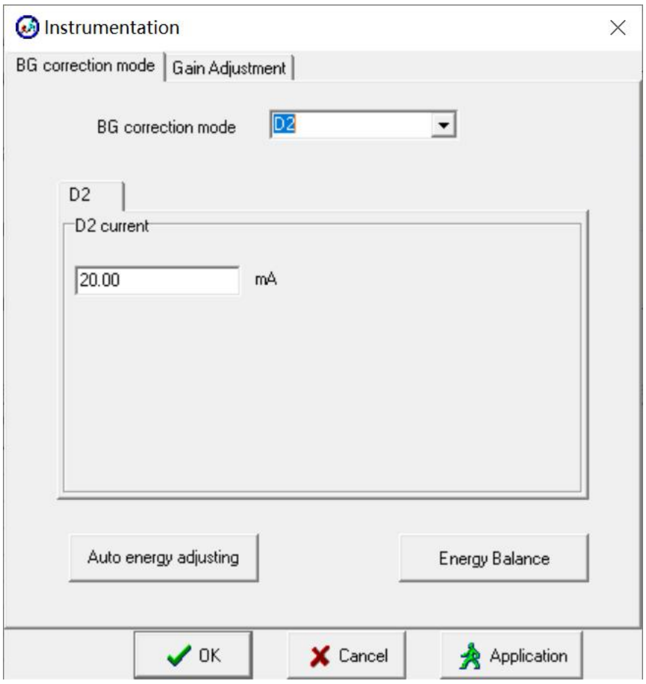
2.4. 9 Background correction mode selection window

2.4. 9.1 Selection of background correction method: change the background correction method

Background correction methods: three;

1、 No
2、 Background correction of deuterium lamp
3、 self absorption

2.4. 9.2 Enter: main menu [instrument] | [background correction].



BG correction mode: D2

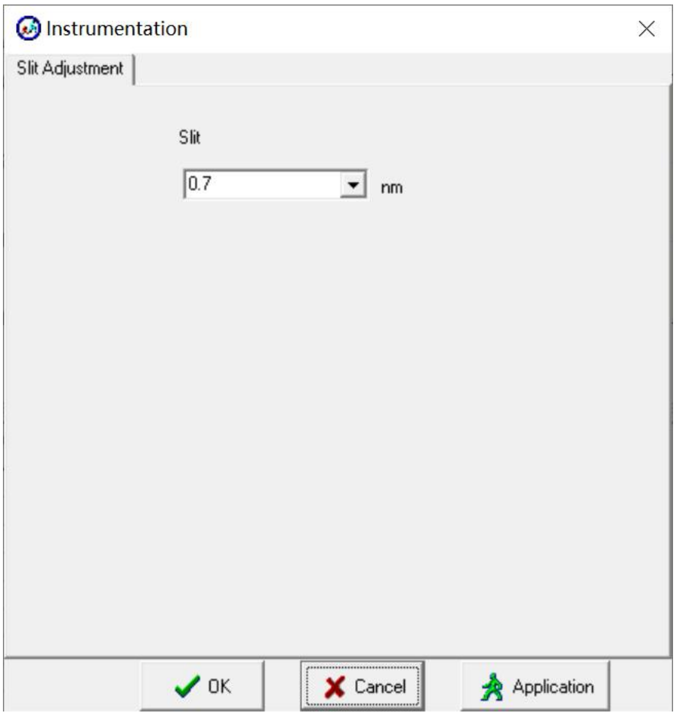
D2 current: 20.00 mA

2.4. 10 Adjust slit window

2.4. 10.1 Slit adjustment content: slit adjustment changes the instrument bandwidth

Slit gear: 0.1 nm 0.2 nm 0.4 nm 0.7 nm 1.4 nm 2 nm

2.4. 10.2 Enter: main menu [instrument] | [adjust slit].

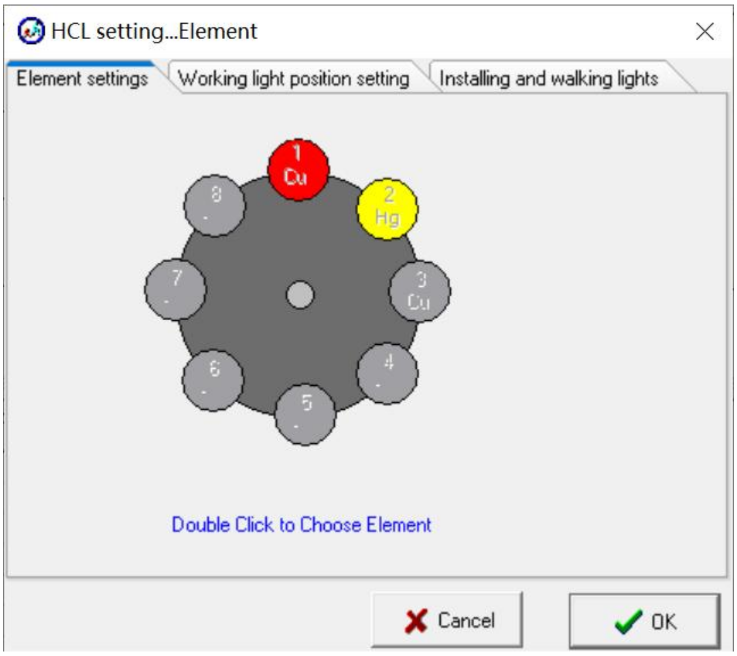


Slit: 0.7 nm

2.4. 11 Automatic element 8 light setting window

2.4. 11.1 Setting content of automatic element lamp: set the name of 8 element lamps and change the position of work lamp and preheating lamp

2.4. 11.2 Enter: main menu [instrument] | [element selection].



2.4.12 Heating Parameter Setting

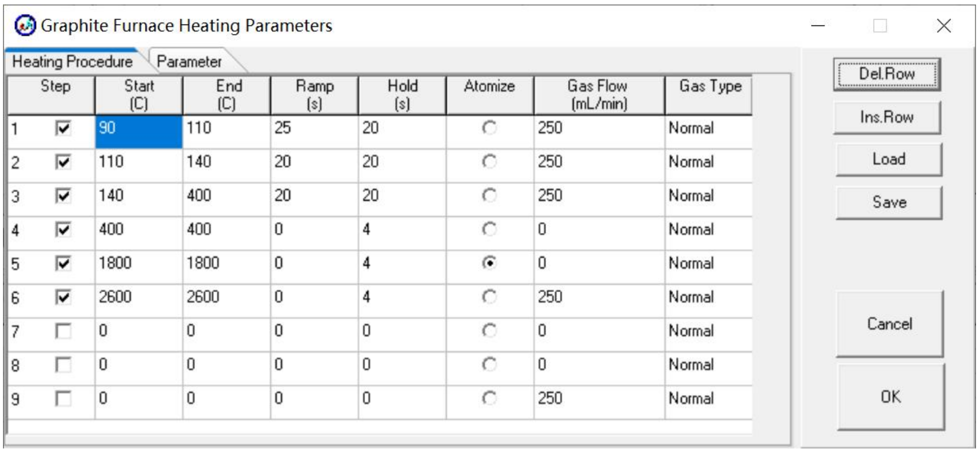
2.4. 12.1 heating parameter setting content: change the temperature rise program of element measurement by graphite furnace method, and the following settings can be made:

1. Start temperature, end temperature, seconds of ramp heating time, maintenance time, atomization and gas stop of each stage of heating procedure.

2. Set atomization temperature rise mode: one of current control, optical control and time control can be set. The time control can set the high-power heating time (unit: 0.1 s), generally 0.4 s.

3. Set water cooling time: generally 30 seconds.

2.4. 12.2 enter: main menu [view] | [graphite furnace] | [graphite furnace power control window] or click "heating parameter setting" in the "instrument conditions" window.



Step	Start (C)	End (C)	Ramp (s)	Hold (s)	Atomize	Gas Flow (mL/min)	Gas Type
1 ☑	90	110	25	20	○	250	Normal
2 ☑	110	140	20	20	○	250	Normal
3 ☑	140	400	20	20	○	250	Normal
4 ☑	400	400	0	4	○	0	Normal
5 ☑	1800	1800	0	4	●	0	Normal
6 ☑	2600	2600	0	4	○	250	Normal
7 ☐	0	0	0	0	○	0	Normal
8 ☐	0	0	0	0	○	0	Normal
9 ☐	0	0	0	0	○	250	Normal

Heating Parameter:

The "graphite furnace heating parameters" window is the main interface for all settings which are related to the operation of the graphite furnace. The page is filled with the recommended conditions for each of the elements. It is strongly recommended to use these settings or to start the program optimization process by carefully modifying these parameters. Once all settings are accepted, confirmed with OK button.

✧ Note Heating Parameters:

It is recommended to use the default parameters for each element. The parameters relate to the graphite tube type (Platform atomization or Wall atomization). Platform atomization is the recommended option for most elements with the exception of the very nonvolatile elements.

If you want to modify the recommended conditions, you may change the temperatures, the ramp, hold time and specific actions during the program sequence in the respective boxes. Program steps are activated by checking the box in the second row of the program page. Only marked steps will be activated. Delete or insert rows by selecting the corresponding buttons.

Be aware that every change to the program may change the conditions of the entire pretreatment and measurement process which may have an influence on accuracy and precision of the determination. Changes to the graphite furnace program require training and experience. In the following section only the individual function choice is explained, physiochemical influence on sample and result of the determination are not include.

- ✧ Start/ End:

These boxes define the starting temperature and the end temperature of a heating process. The temperatures relate to electrical parameters in the instrument. The parameters required to reach e.g. 120°C in the program, featured above are set immediately once the program is activated. The "start" and the "end" temperature relate to the "ramp time" which defines a linear increase of the electrical settings from the starting point to the end point, in this case from 120°C to 150°C

- ✧ Step:

Different stages of the heating parameter. Only the steps which are selected will be executed.

- ✧ Ramp(s):

The time needed to heat from the starting point to the end point (see above).

- ✧ Hold(s):

Time selected to retain the end temperature until the next step is activated. E.g. if 3 seconds is set, after reaching the set temperature, the furnace will remain at this temperature for 3 seconds before executing the next step.

- ✧ Atomize:

If selected, the step is defined as the atomization process. The absorbance will be read right from the beginning of the atomization. Modifications to read/ integration time can be made in the "instrumentation page" as required.

- ✧ Gas Flow:

Gaseous residues from the samples are removed from the furnace with a purge gas flow from both sides of the tube towards the center and out of the dosing hole. This function should be activated during the steps where matrix is removed from the tube, i.e. during drying, pyrolysis and heating out. In all these cases the flow through the furnace should be maximum, i.e. each 250 mL/min Ar from both sides. Shortly before and during atomization this flow should be stopped to ease down the atmosphere inside the tube.

In some special and rare cases, a small flow through the furnace in the atomization step is beneficial to reduce the residence time of analyze and matrix during the read cycle. The option for this step is a flow of 50 mL/min from both sides. Make sure to use 2 steps with reduced flow as described in the case of "stop flow".

- ✧ AZ Temperature Ramp Mode:

Setting the electrical parameters to the desired final temperature will result in a slow temperature increase with an asymptotic approach to the set temperature. The function "ramp" in the "ramp to atomization" window featured above is usually not suited to obtain best atomization conditions. Instead the instrument is equipped with a detector controlled fast heating. This "automatic" function is the default setting and recommended to use. When the atomization process starts, the tube is heated with the maximum power until the temperature reaches the respective setting. An optical sensor controls this process to keep the set temperature. Atomization processes usually keep the temperature between 1500°C and 2500°C. In this range the fast heating process runs with a speed of approximately 1.3 °C /ms. Ramping from 1000°C (in the pyrolysis step) to 2500°C in the atomization step will take about 1s. This time required for a certain temperature gap can be roughly estimated and set with the function "time controlled" in steps of 0.1s. Using this function requires experience and may shorten tube lifetime substantially and should only be operated by experienced users under special conditions.

✧ Graphite Tube:

make sure the recommended tube type for the intended determination(s) is installed (see section above). In most cases the platform tube should be used. Open the furnace with the respective button. In order to align the dosing hole orthogonally and the light beam's position, they should be fixed by using the alignment tool; then close the furnace. New tubes always must be in condition in order to adjust to the fast ramps and high temperatures. It is t mandatory to perform the "Graphite tube aging" for every new tube inserted into the furnace. This action will significantly prolong the lifetime of the graphite tube. The tube temperature must be memorized on the optical sensor. This is processed with the function "temp calibration". After these procedures the tube is ready for the analytical work.

✧ Number of Firings:

this function counts the number of atomization cycles performed with the tube. Depending on elements measured, temperature program set and matrix present in the sample, the typical lifetimes of a tube ranges from 300-500 cycles. This function is an indication for analytical quality control.

✧ Cooling Time(S):

The time for the furnace to cool down after heating is selected with this function. It is recommended to keep default setting which is 30s. Shorter time may be possible if volatile elements are atomized and heated out at temperatures below 2200°C.

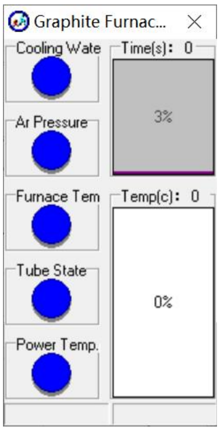
2.4. 13 working status window of graphite furnace power supply

2.4. 13.1 contents displayed in the working status window of graphite furnace power supply:

1. Display the time, temperature and number of stages of the current stage in the heating process.

2. Display the cooling water flow of graphite furnace power supply, protective gas pressure, furnace body temperature alarm and graphite tube installation status, The green light indicates normal, and the red light indicates alarm.

2.4. 13.2 Enter: display automatically during measurement, or main menu [view] | [graphite furnace] | [graphite furnace status window]



The status of the safety interlocks for the graphite furnace are displayed on the furnace control box. It includes pressure and flow rate of cooling water, pressure of inert gas, furnace temperature and integrity of the graphite tube. A red-colored icon indicates a failure situation. The window also shows the status of the current temperature and the percentage progress of the program cycle based on total time.

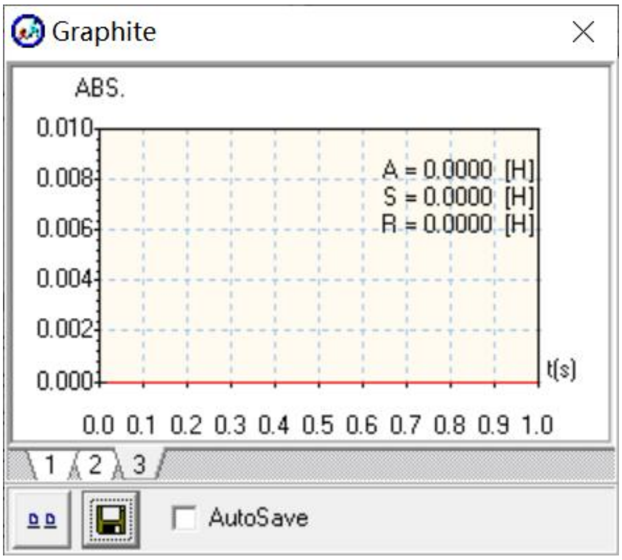
2.4. 14 Atomized waveform window

2.4. 14.1 contents displayed in atomization waveform window:

1. Display the atomization pattern and superposition diagram of several waveforms measured by graphite furnace method.

2. Press "⧉" button to display superimposed graphics, and press "💾" button to save atomized graphics.

2.4. 14.2 access: display automatically during measurement, or main menu [view] | [graphite furnace] | [atomization graphics window]

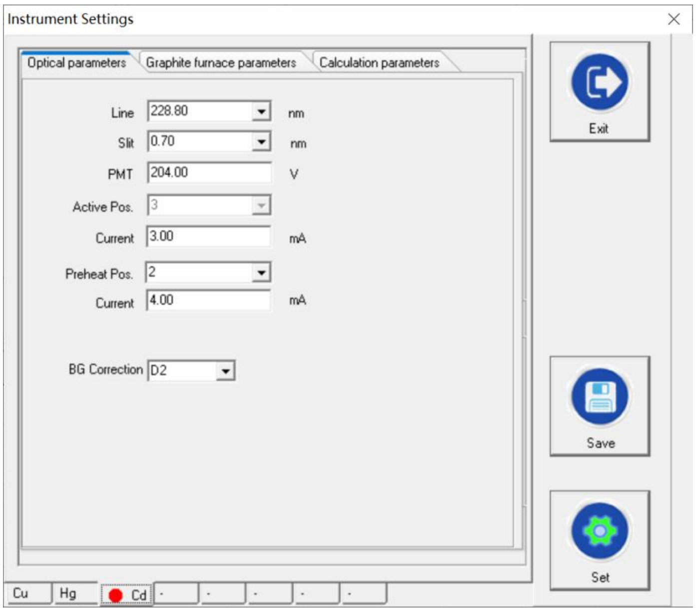


A = 0.0000 [H]
S = 0.0000 [H]
R = 0.0000 [H]

2.4. 15 Instrument condition window

2.4. 15.1 Enter: press the " Instr.Set " button, or the main menu [instrument] | [condition setting]

2.4. 15.2 contents displayed in the optical parameter window of instrument conditions:



Line 228.80 nm
Slit 0.70 nm
PMT 204.00 V
Active Pos. 3
Current 3.00 mA
Preheat Pos. 2
Current 4.00 mA
BG Correction D2

Line: selection of the analytical line. The default setting is the most sensitive line of the element (primary resonance line). Others can be chosen from the drop box which are usually less sensitive that extend the working range of the instrument to higher concentrations. In rare cases, such as Pb which the default line is not the most sensitive, but it provides best signal to noise for complex samples.

Slit: choose the spectral bandwidth. Options are 0.1 nm, 0.2 nm,0.7 nm and 1.4 nm. The default value for the respective element will provide the best signal to noise ratio and the widest dynamic working range.

PMT: The PMT voltage of the photomultiplier tube, is set automatically. Changes to this setting is not recommended.

Active Pos.: The working lamp position is indicated here. The user may change the active position. Preheat pos.: Indication of the lamp position for pre-heating. Changes should be made in the lamp window only.

Meas. Mode: This window defines the mathematical operation with which the data from the detector are handled. “peak area” should be selected for Graphite furnace AAS measurements. In some cases “peak height” can be used as alternative in GF-AAS. The values recommended above are the default settings.

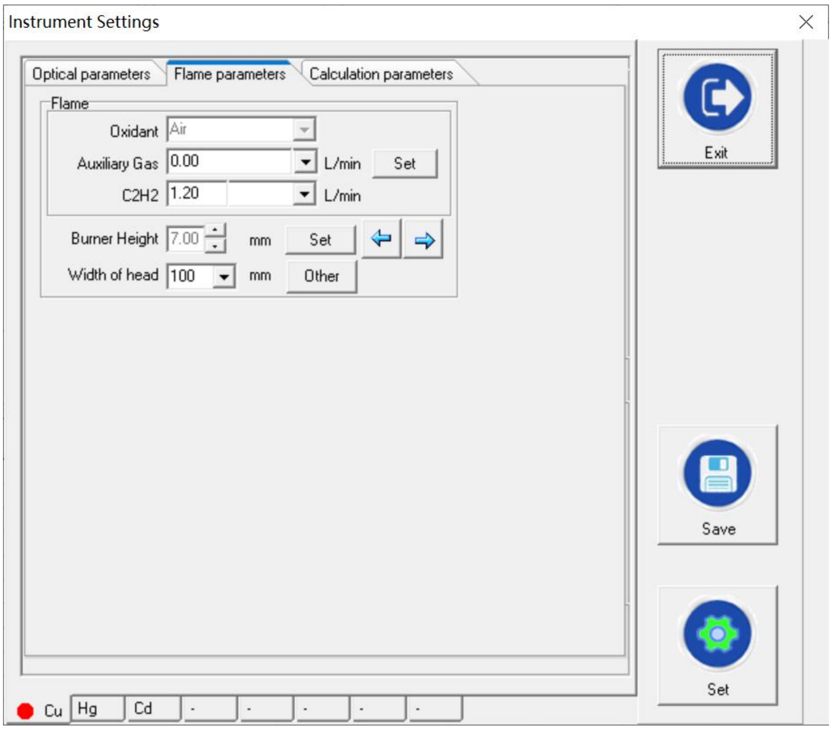
Replicates Mode: manual or auto. In “manual” operation the sample is injected by hand with a pipette. Auto means automatic injection with the sampler. Usually the auto sampler will be used for graphite furnace analysis.

Current: Allows to change the lamp current. It is strongly recommended to use the default parameters.

Background Correction: D2 lamp or S-H background correction can be selected

Spec.Mode: Absorption and Emission (AAS or AES) can be selected. Measurements in flame emission require adapted operation of the instrument.

2.4.15.3 instrument condition flame parameter:



Instrument Settings

Flame

Oxidant: Air

Auxiliary Gas: 0.00 L/min

C2H2: 1.20 L/min

Burner Height: 7.00 mm

Width of head: 100 mm

Oxidant:

Choose between Air or N2O

Oxidant Flow:

Flow rate of the additional oxidant gas. This function will increase the fixed oxidant flow through the nebulizer (~6L/min) by the value indicated in the drop box. The function is predominantly used, if energy-rich organic solvents, such as e.g. kerosene, are aspirated.

C2H2 Flow:

Flow rate of ethyne

Burner Height:

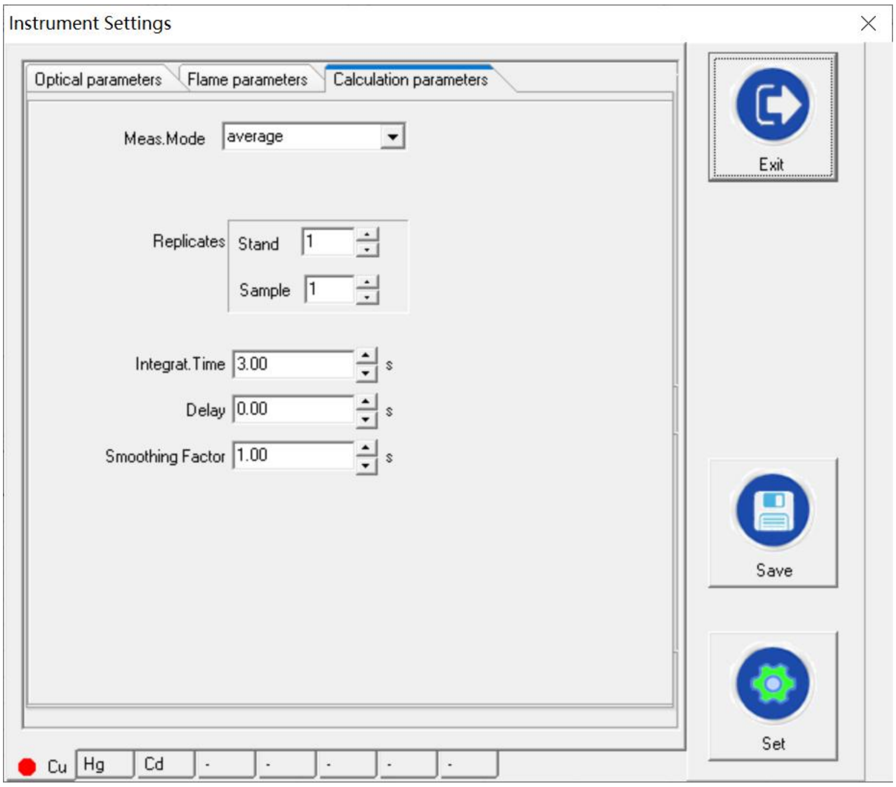
The distance between burner head and light beam can be adjusted from 5 mm to 25 mm.

Burner Type:

select the burner type, 50 mm or 100 mm. The 100 mm burner provides best sensitivity and detection limits for the air- ethyne flame. The 50 mm burner head can be used for air- ethyne operation as well. It will reduce sensitivity by about 1.7 x and thus extend the dynamic range to higher concentrations. Nitrous oxide- ethyne flames can only be operated with the 50 mm burner head.

Activate the “Other” button located on the right side to enable the scraper setting. (The scraper should always be used during the analysis using N2O-C2H2 flame). An interval of 60 s will be enough to run standard N2O-C2H2 flames with high long-term stability.

2.4.15.4 Instrument condition calculation parameter:


Instrument Settings

Meas.Mode: average

Replicates
Stand: 1
Sample: 1

Integrat.Time: 3.00 s

Delay: 0.00 s

Smoothing Factor: 1.00 s

Replicates Sample:

This selection defines the number of repetitive measurements for samples.

Replicates Std.:

This selection defines the number of repetitive measurements for blank and standards.

Integration Time:

The instruments is collecting a complete set of data each 20 ms. These are summed up for a certain amount of time which is defined with this function. A typical read time for a graphite furnace analysis is 3-5 s. The individual data are evaluated in various modes, are described above under the function “measurement mode”.

Delay:

Data collection is retarded by a certain amount of time in order to stabilize the analytical absorbance. In graphite furnace AAS only experienced users should apply this function.

Smoothing Factor:

No smoothing should be used in GFAAS. After all instrument settings are executed and automatic peak picking finishes, Close the scan WL window, and click to set standard and sample parameters.

Chapter 3 Element measurement

3.1 standard curve method

3．1．1 Principle

First prepare a series of standard solutions with different concentrations, under the same conditions as the specimen, measure their absorbance separately, and map the absorbance with the corresponding concentration, and the resulting straight line is called the standard curve or working curve.

The absorbance of the specimen is then determined, and the concentration of the specimen is found from the working curve.

Regression analysis is used for data processing for standard curve quorum calculations in the computer. The principle for determining the regression line is to make it the sum of squares of the errors with all experimental points to the minimum.

There are 6 kinds of regression equations：

1. A=k1C+k0
2. Pass Zero A=k1C
3. A=k2C^2+k1C+k0
4. Pass Zero A=k2C^2+k1C
5. Nonlinear two-coefficient equation
6. Nonlinear three-coefficient equation

The standard solution containing the elements to be measured is added to 3 to 4 containers in the prescribed proportional order, and then diluted the above solution to the specified volume with the same solvent. Figure 3.1. (1) Indicates the concentration of this set of sample solutions prepared.

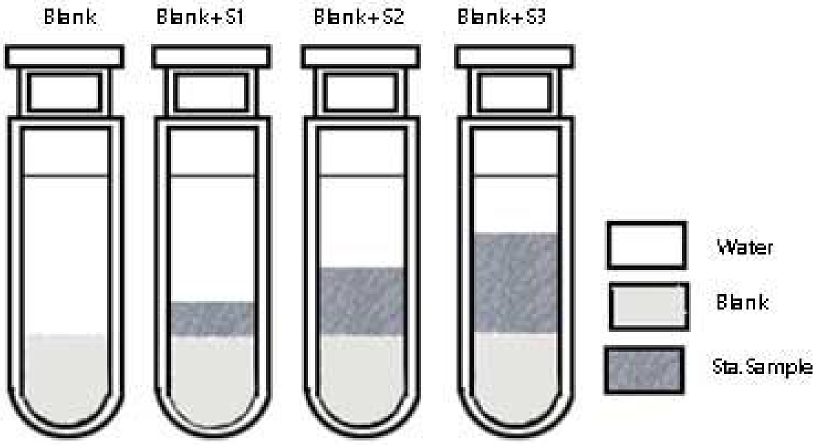
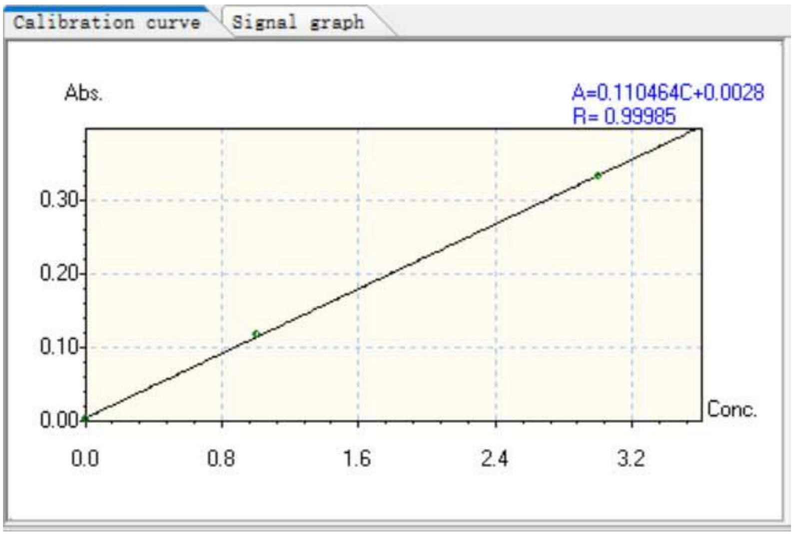


Figure 3.1. (1) Sample preparation by standard curve method

This set of samples is subjected to a pipetting assay and the absorbance of the blank solution is measured. The absorbance of each solution is plotted, that is, the longitudinal axis represents the absorbance, and the horizontal axis represents the standard concentration.

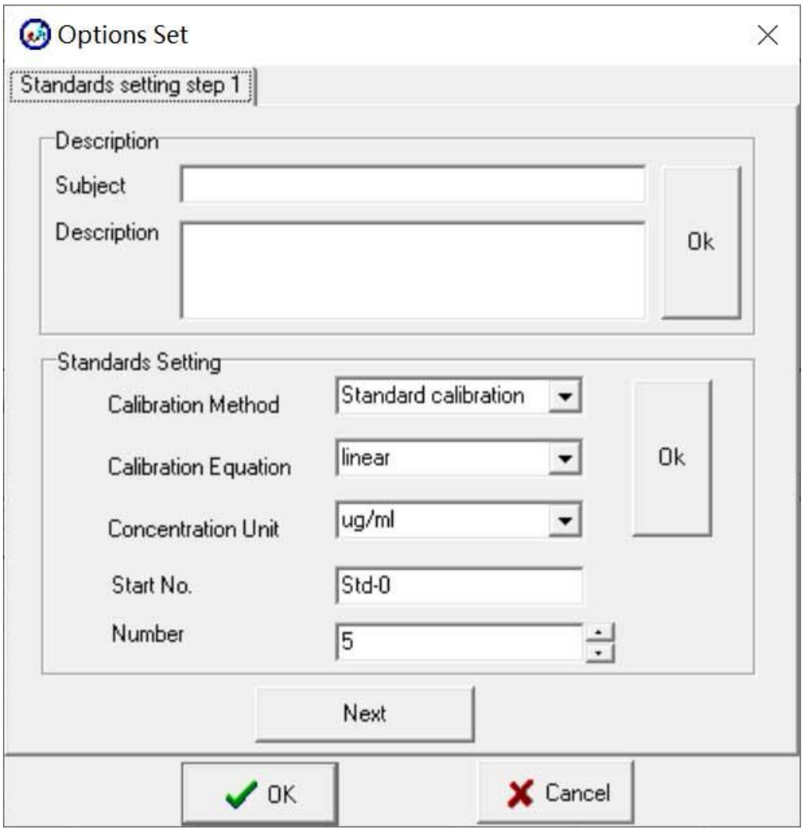


A=0.110464C+0.0028
R= 0.99985

Figure 3.1. (2) The principle of the standard curve method

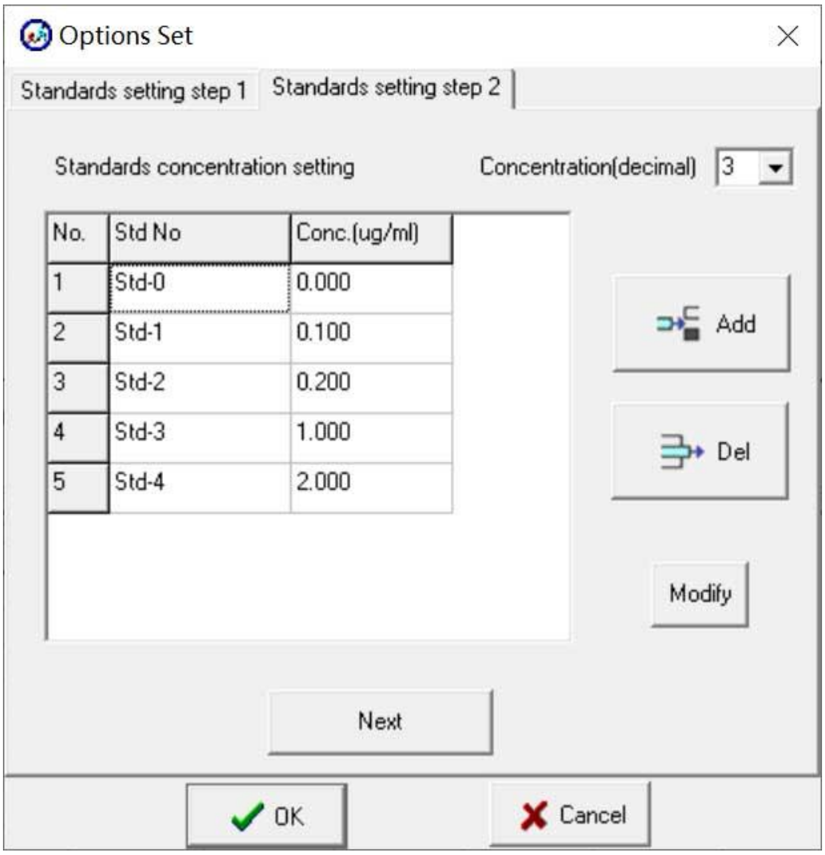
3．1． 2 Step

Step 1: Click the [Sample] tool, select the standard curve method for correction method, select the correction equation [1-6] for: 6 [Nonlinear three-coefficient equation]。



Subject
Description
Calibration Method: Standard calibration
Calibration Equation: linear
Concentration Unit: ug/ml
Start No.: Std-0
Number: 5

Step 2：Press Next to fill in the standard solution concentration

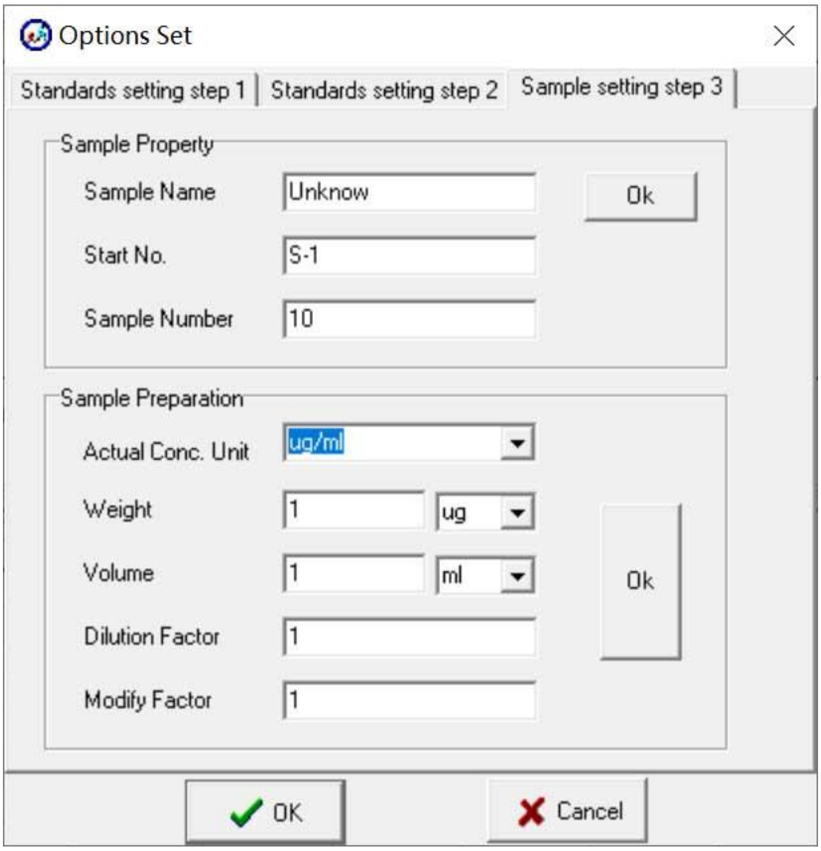


Standards concentration setting

Concentration(decimal) 3

No.	Std No	Conc.(ug/ml)
1	Std-0	0.000
2	Std-1	0.100
3	Std-2	0.200
4	Std-3	1.000
5	Std-4	2.000

Step 3：Press Next to perform sample setup and confirm exit



Sample Property

Sample Name: Unknow

Start No.: S-1

Sample Number: 10

Sample Preparation

Actual Conc. Unit: ug/ml

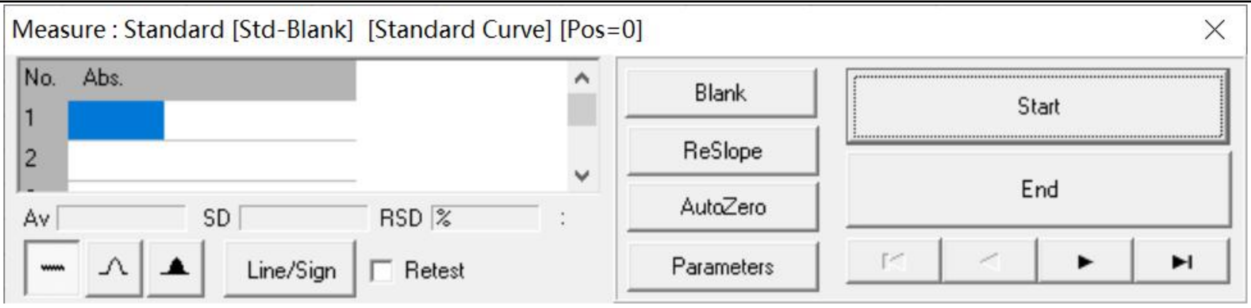
Weight: 1 ug

Volume: 1 ml

Dilution Factor: 1

Modify Factor: 1

Step 4：Press Measure to enter the measurement control interface.



Step 5: suck and spray blank solution or distilled water to zero.

Step 6: suck and spray the blank solution, sample, sample + S1, sample + S2... Measure the concentration of the standard sample and draw the standard curve at the same time. After measuring all the prepared solutions, the coordinates of the standard curve can be adjusted automatically.

Step 7: suck and spray the sample to be tested, and the concentration can be read out directly.

Step 8: change the "correction equation" afterwards: Step 1 change in the interface, click Modify, and then click "Cancel" to exit!

Step 9: data saving and printing.

3.2 Standard addition method

３．２．1 Principle

Transfer the same volume of sample solution to be tested into 3 ~ 4 containers, and then transfer the standard solution containing the element to be tested The solution shall be added to the container in the specified proportion sequence, but one container shall be reserved without standard solution. Then use the same solvent Dilute the above solutions to the same volume respectively. It should be noted that the blank solution should also be diluted as other parts Dilute. Figure 3.2 1 is the concentration diagram of the prepared sample solution.

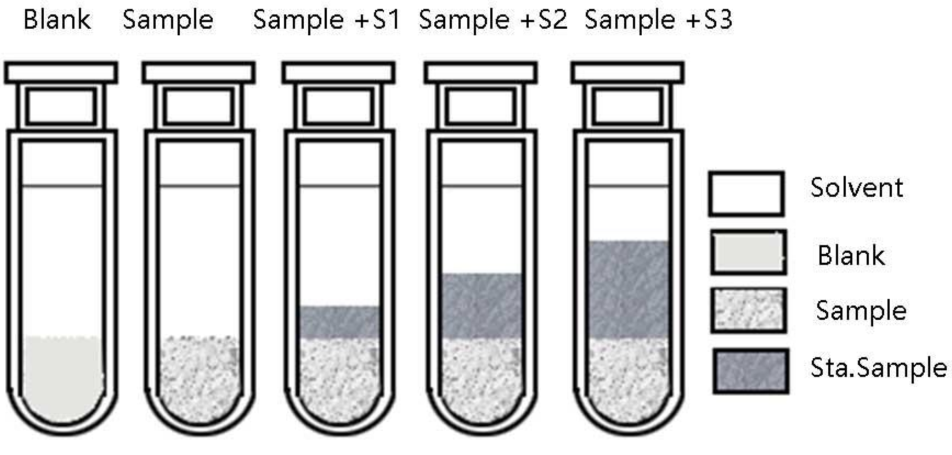


Figure 3.2 1

This set of samples is subjected to a pipetting assay and the absorbance of the blank solution is measured. The absorbance of each solution is plotted, that is, the longitudinal axis represents the absorbance, and the horizontal axis represents the standard equivalent concentration added.

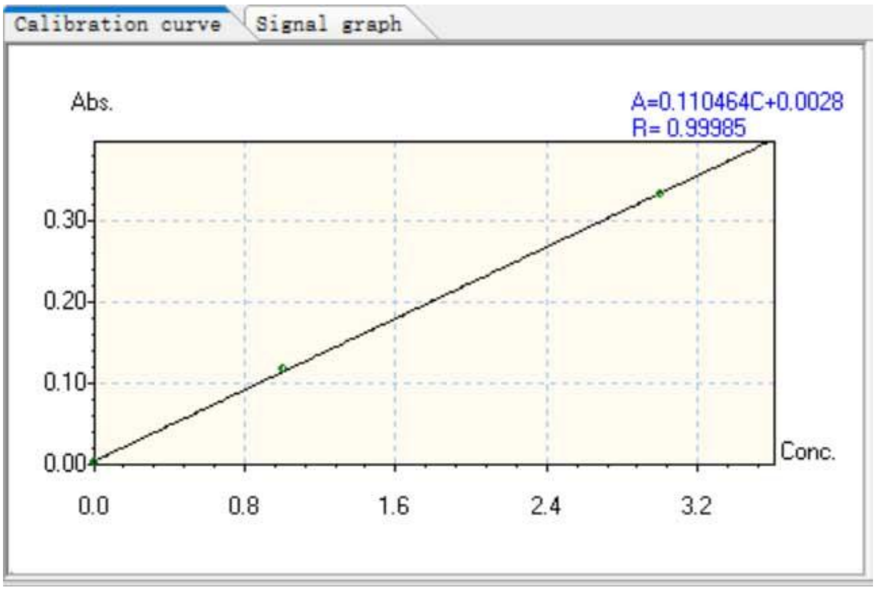


Figure 3.2 2

At this time, in this group of solutions, the amount of co-existing substances is exactly the same, and the obtained calibration curve should be a straight line, and its slope is different from the slope of the calibration curve made of pure standard solution, representing the degree of matrix interference. This standard curve does not pass through the origin, and if it is assumed to be linear in the low concentration range, this line can be extrapolated to the absorbance of the blank solution. At this time, the sample solution without the standard solution, that is, the diluted concentration of the sample to be measured, can be calculated (Figure 3.2.2).

This method applies to samples where there is a matrix interference and the composition of the matrix is unknown, and the same interference cannot be added to the standard solution.

The use of standard accession must be noted:

(1) The part of the correction curve that is linear should be measured.

(2) The standard concentration added should be 2 to 5 times the concentration of the sample, so that the slope of the correction curve is close to 1 to obtain higher measurement accuracy.

(3) The difference between the slope of the calibration curve of this method and the slope of the correction curve prepared by pure standards should not exceed 20%.

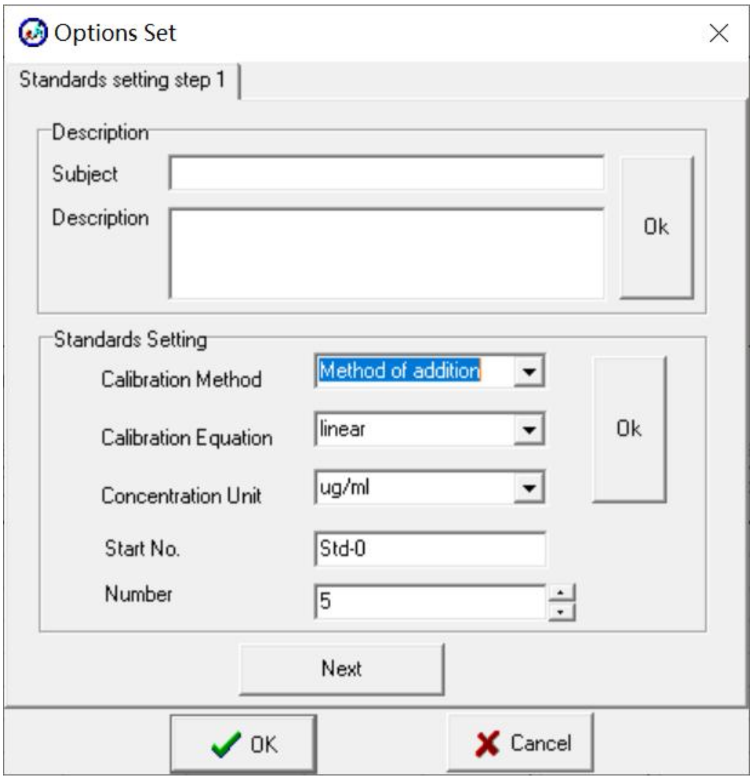
If it exceeds 20%, indicating that the interference is too severe, the accuracy of the analysis will be reduced. Therefore, the co-existing substances should be removed in advance before the assay is carried out。

If a batch of samples to be measured has a very similar matrix composition, that is, the interference of the substrate to be measured element is completely consistent, the remaining samples can be determined using the above calibration curve without having to prepare each sample into a group of samples. This method is called the simple standard addition method. When using the simple standard addition method, the calibration curve should still be prepared into a group of solutions according to the above method, one of the samples to be measured, and the slope and intercept of the calibration curve should be determined.

The standard addition curve can be converted into a normal standard curve, and the concentration of other samples can be read directly。

3．2． 2 Test step

Step 1: Click the [Sample] tool, select the standard addition method for the correction method, select "A=k1C+k0" for the correction equation, and the remaining steps (same as 3.1)。



Calibration Method: Method of addition
Calibration Equation: linear
Concentration Unit: ug/ml
Start No.: Std-0
Number: 5

Step 2: Pipetting blank solution to determine absorbance. Sequential determination: sample, sample +S1, sample +S2..., after measuring all the configured solutions, at this time jump out of the dialog box, showing the solubility of the substrate sample, while asking whether it is converted to a standard curve.

Step 3: Confirm that the standard addition curve can be converted to a standard curve.

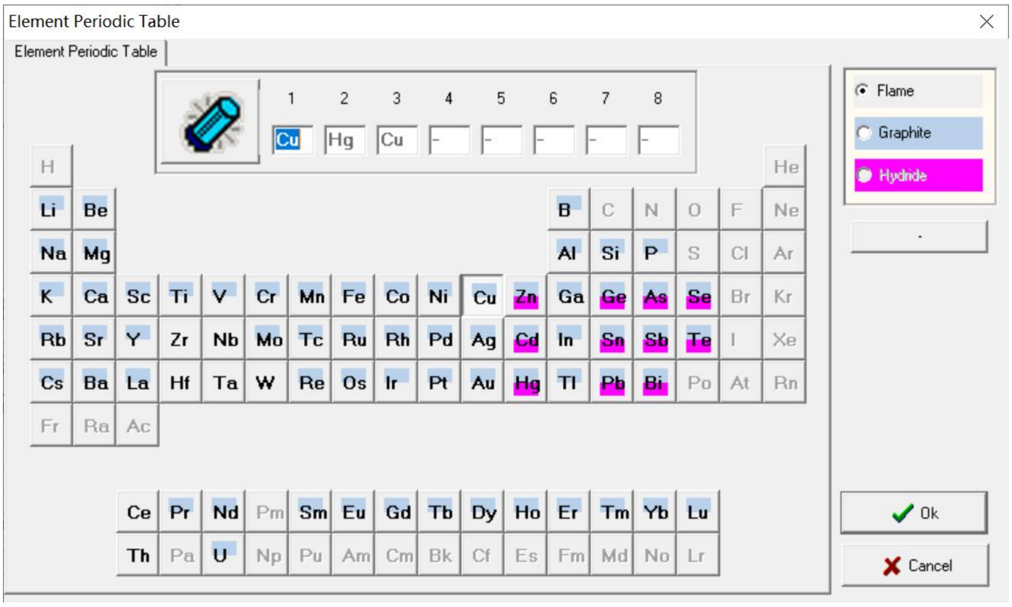
Step 4: Pipetting other samples and reading the concentration directly.

3.3 Flame measurement

3.3.1 Preparation

1. Select flame measurement mode: When element selection, in the periodic table window, the measurement mode selects "Flame" Action: Main menu [Instrument] | [Element Selection]

Then double-click the light to select the element：



● Flame
○ Graphite
○ Hydride

1 Cu
2 Hg
3 Cu
4 -
5 -
6 -
7 -
8 -

Finally, the condition setting: the main menu [Instrument] | [Condition Setting] confirm.

2. Using the flame method, please carefully check the gas path and water seal before entering the measurement. When you are sure that you are correct, you can select the main menu [Instrument] | [Ignition] or click the tool button " On " to ignite the flame.

If you think the flame is too large, too small, or the flame is not in a reasonable position, you can use the up-down, left-right adjustment knobs of the burner to adjust the position of the burner, and use the flow adjustment knobs of the gas and gas auxiliary gas to adjust the flame size until it is adjusted to the best state. You can then select the main menu [Measurement] | [Start] to open the measurement window.

3.3.2 Steps to measure elements using the flame method: Please read the 3.1 standard curve method and the 3.2 standard addition method carefully.

3.4 Graphite furnace measurement

When you need high sensitivity measurement, you can use graphite furnace method to determine the sample.

3.4.1 Preparation before graphite measurement

3.4.1.1. Installation and adjustment of graphite furnace bodies

1. Remove the flame burner
2. Set the instrument according to the steps of "3.4.2.1 instrument condition setting", and find the wavelength peak of the measuring element.
3. Install the graphite furnace body and the graphite tube at the same time
4. Use the up, down, left and right adjustment knobs of the atomizer to adjust the position of the graphite furnace body until the energy of the sample channel is in the best state.

3.4.1.2. Check the supporting facilities of graphite furnace power supply

Unlike the flame method, the graphite furnace method does not require ignition, but requires the connection of the gas circuit to the waterway. Before entering the measurement, please carefully check the gas and waterways to avoid leakage.

Before measuring, ensure that the gas path, waterway, circuit and communication line of the graphite furnace power supply and the atomic absorption host are connected normally, and the instrument power supply is turned on.

3.4.1.3. Select the flame measurement mode:

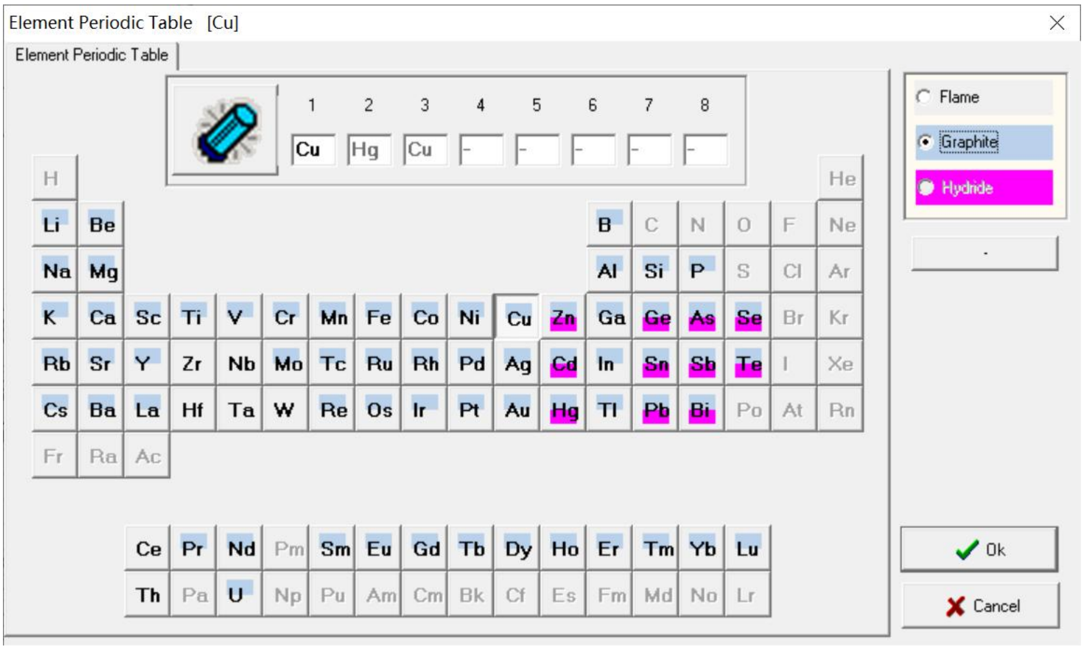
When selecting elements, in the periodic table window, the measurement mode selects "Graphite Furnace"

3.4.2 Elemental operation steps are measured using the graphite furnace method

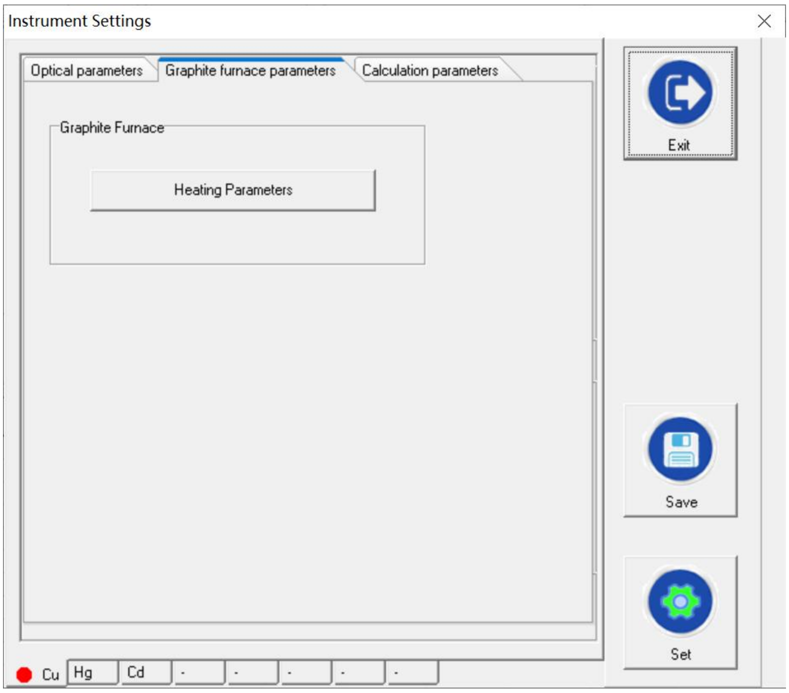
3.4.2.1 Instrument condition setting of graphite furnace

Step 1: Select the element, the main menu [Instrument] | [Condition Settings] or click the Tool button "Instr.Set"

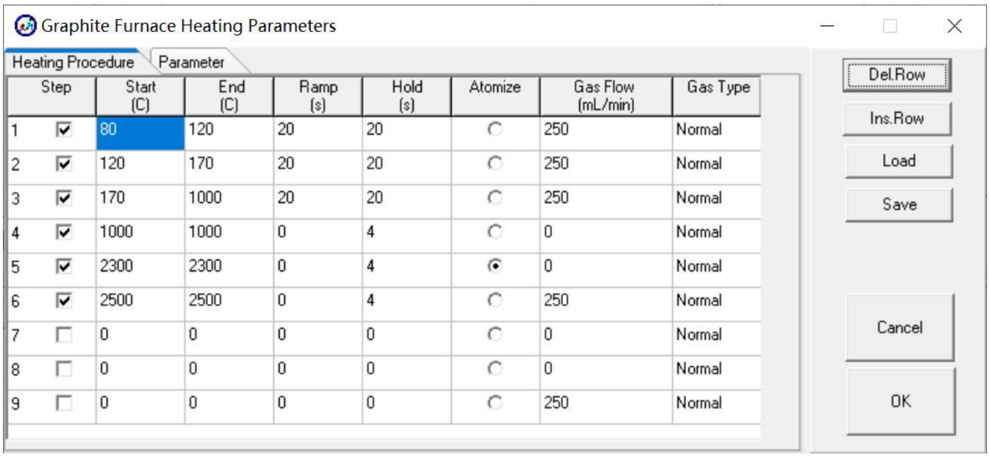
Enter the Element Light Settings window: Press OK to enter Condition Settings. Then double-click on the lamp to select the elements: when the elements are selected, in the periodic table window, the measurement mode selects "Graphite Furnace".



Step 2: Condition Settings:



Step 1. Heating parameter settings: click "Heating Parameter " to enter the "Heating Parameter Settings" window

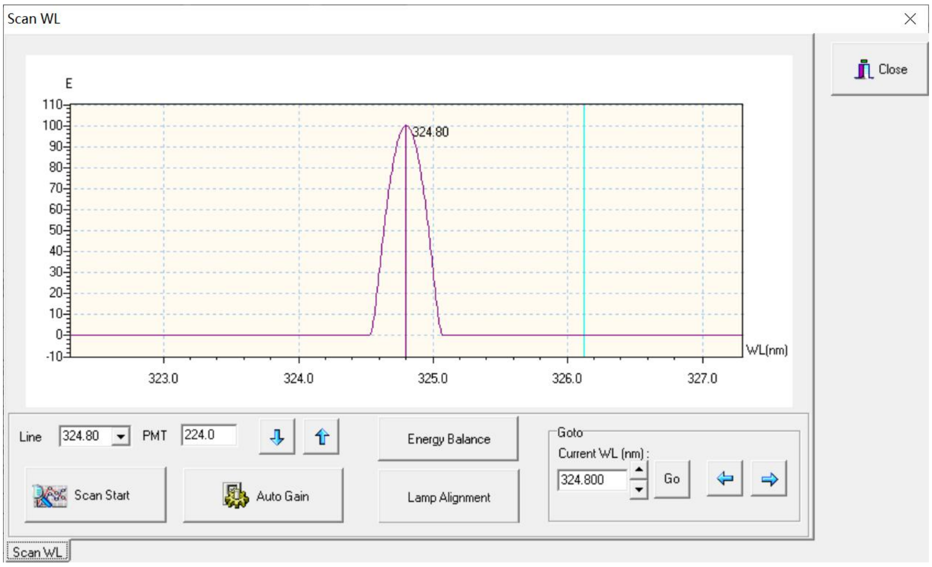


Step		Start (C)	End (C)	Ramp (s)	Hold (s)	Atomize	Gas Flow (mL/min)	Gas Type
1	☑	80	120	20	20	○	250	Normal
2	☑	120	170	20	20	○	250	Normal
3	☑	170	1000	20	20	○	250	Normal
4	☑	1000	1000	0	4	○	0	Normal
5	☑	2300	2300	0	4	●	0	Normal
6	☑	2500	2500	0	4	○	250	Normal
7	☐	0	0	0	0	○	0	Normal
8	☐	0	0	0	0	○	0	Normal
9	☐	0	0	0	0	○	250	Normal

Fig. 3.4.2.1-4 Heating parameter settings

Step 2. After modifying the conditions, click the "OK" key to automatically set the wavelength slit lamp current photomultiplier tube voltage, and enter the wavelength peaking interface; Or click the "Auto Peak" key and the software automatically completes the peak hunt (step 3 can be omitted) directly to step 4。

Step 3: Wavelength Peaking: Click "Rescan" key, automatic wavelength peaking, find the wavelength position of the analysis line, after the scan can be used "Automatic adjustment of energy" button, automatic adjustment of high pressure, so that the current energy to reach the best; If the wavelength peak is not ideal, repeat step 4 until the analysis line wavelength position is optimal and the energy is most appropriate.



Line 324.80 PMT 224.0

Current WL (nm): 324.800

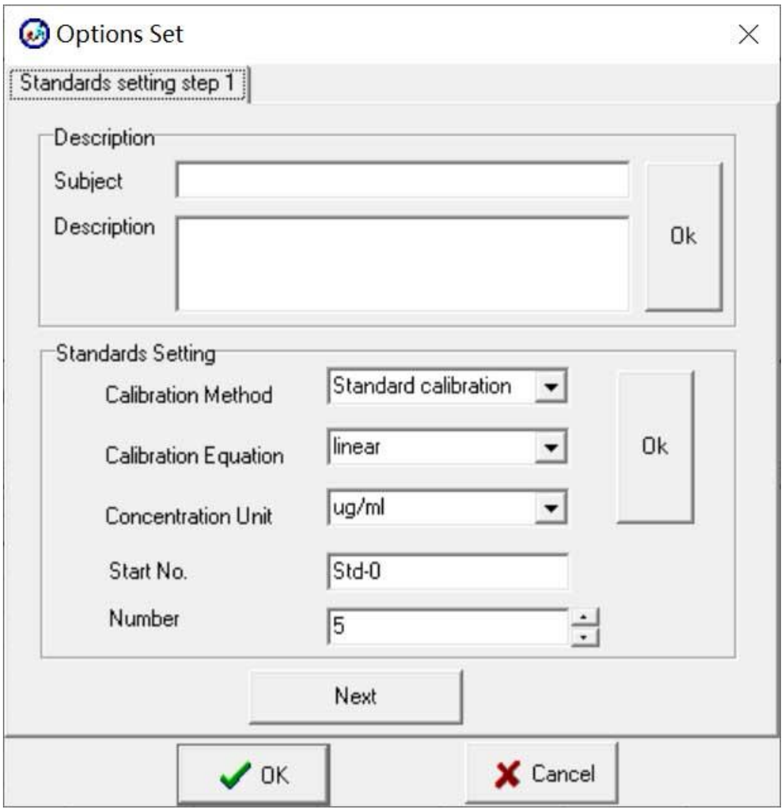
Fig. 3.4.2.1-5 wavelength peaking

Click "Lamp Position Auto Fine-tune" to make the light position automatically fine-tuned to the optimal position。

Step 4: Carefully adjust the position of the hollow cathode lamp so that the reading of the light energy bar on the screen is the maximum, observe the high voltage of the photomultiplier tube, and the wavelength scanning. Step 3 can also be repeated until the analysis line wavelength is optimal and the energy is most appropriate. Click the "Close" key to exit and enter the main interface.

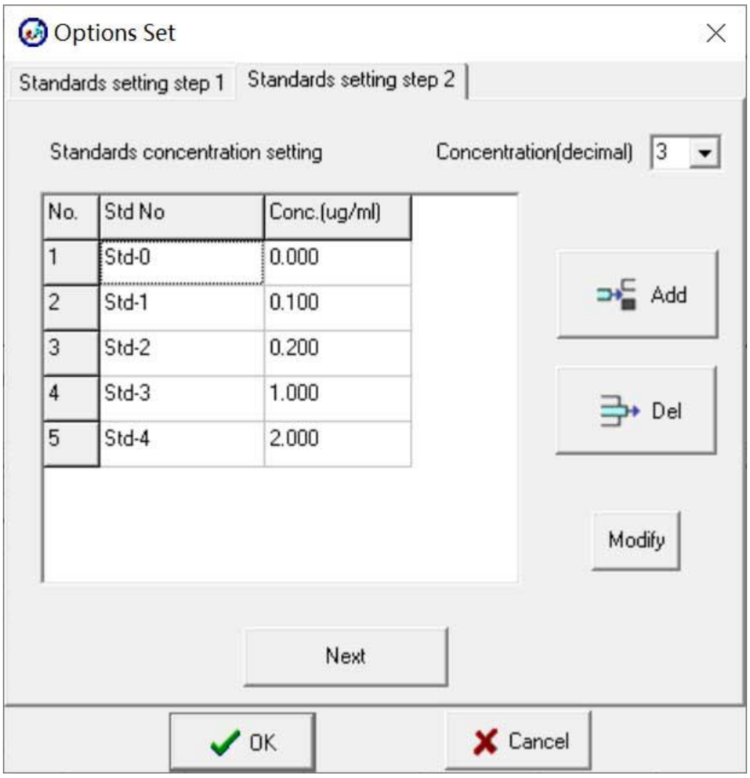
3.4.2.2 Graphite furnace sample condition settings

Step 5: Click Std. Set to enter the standard sample setup line window. Select the calibration curve's "Calibration Method", "Correction Equation", "Concentration Unit", "Standard Sample Start Number", "Standard Sample Quantity" and other settings. Press Next to go to other settings.



Calibration Method: Standard calibration
Calibration Equation: linear
Concentration Unit: ug/ml
Start No.: Std-0
Number: 5

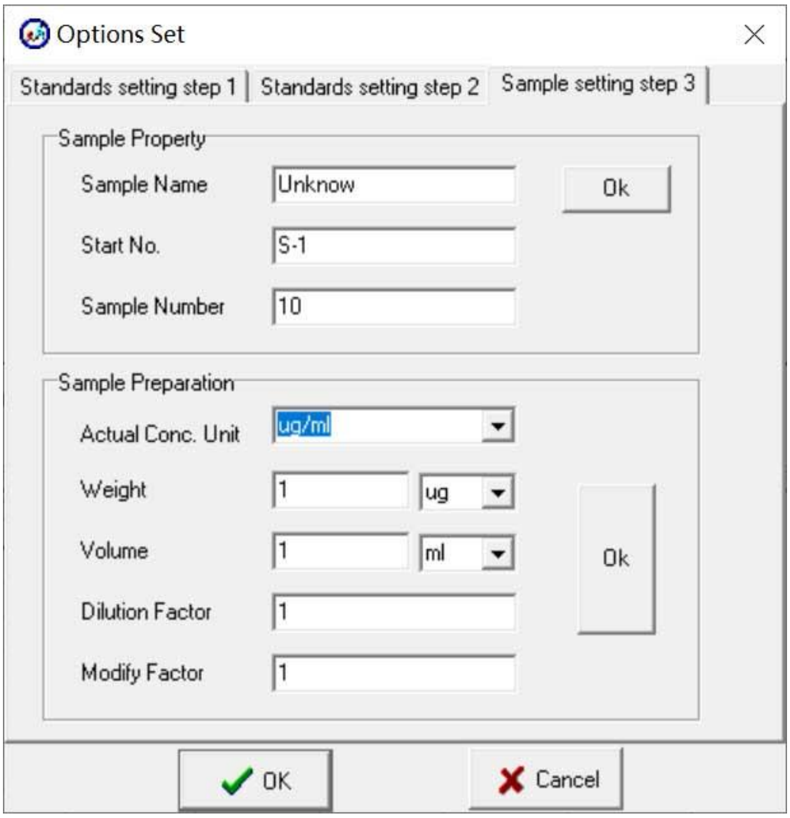
Step 6: Fill in the concentration of each standard point and press Next to enter the other settings.



Concentration(decimal): 3

No.	Std No	Conc.(ug/ml)
1	Std-0	0.000
2	Std-1	0.100
3	Std-2	0.200
4	Std-3	1.000
5	Std-4	2.000

Step 7: Perform "Sample Setup": Fill in the sample properties and sample settings in turn; Press OK to submit, enter the main interface, and the data in the table area will be refreshed to empty.



Sample Property

Sample Name: Unknow

Start No.: S-1

Sample Number: 10

Sample Preparation

Actual Conc. Unit: ug/ml

Weight: 1 ug

Volume: 1 ml

Dilution Factor: 1

Modify Factor: 1

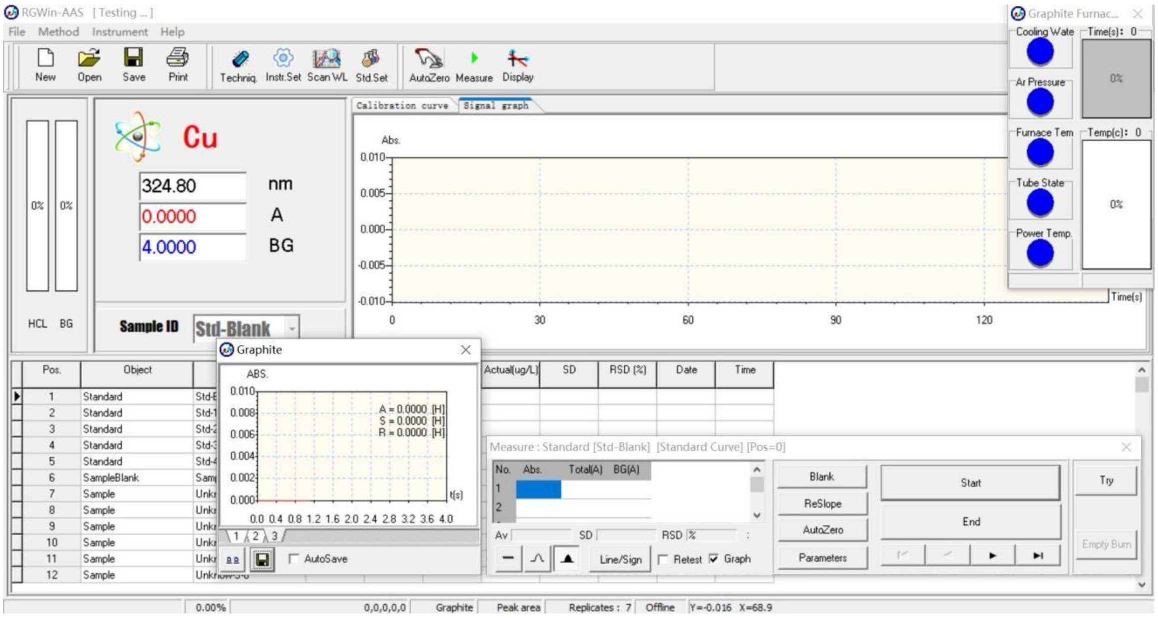
3.4.2.3 Graphite furnace sample measurement

Step 9: Make sure that the gas circuits, waterways, circuits, and communication lines of the graphite furnace power supply and the atomic absorption host are connected normally, and that the graphite furnace power supply is turned on.

Step 10: Zero: Click AutoZero to Zero.

Step 11: Heating parameter setting: If the graphite furnace power supply is not turned on when the conditions are set, the graphite furnace power supply has not received the setting parameters and can be reset again; Or the main menu [View] | [Graphite Furnace] | [Graphite Furnace Power Control Window] to enter the "Heating Parameter Settings".

Step 12: Click Measure to enter the measurement window to take the measurement. The current sample number is displayed in the window header.



324.80 nm

0.0000 A

4.0000 BG

Sample ID: Std-Blank

A = 0.0000 [H]
S = 0.0000 [H]
R = 0.0000 [H]

Pos.	Object	
1	Standard	Std-E
2	Standard	Std-1
3	Standard	Std-2
4	Standard	Std-3
5	Standard	Std-4
6	SampleBlank	Sam
7	Sample	Unkr
8	Sample	Unkr
9	Sample	Unkr
10	Sample	Unkr
11	Sample	Unkr
12	Sample	Unknow-5-6

Actual(ug/L)	SD	RSD (%)	Date	Time

Measure : Standard [Std-Blank] [Standard Curve] [Pos=0]

No.	Abs.	Total(A)	BG(A)
1			
2			

0.00% | 0,0,0,0,0 | Graphite | Peak area | Replicates : 7 | Offline | Y=-0.016 X=68.9

Step 13: Test the heating procedure: Press Test button.

Step 14: Empty Burn Cleaning: Press Empty Burn button.

Step 15: Sample Measurement: Press Start key to make a single measurement, the standard curve is updated in real time, and the sample concentration is calculated in real time.

Step 16: Blank correction and slope recorrection are available when performing sample measurements.

Step 17: Save atomized graphics: In the Atomized Graphics window, press "ꝋꝋ" button to display the overlay graphic, press "💾" button to save the atomized graphic.

Step 18: Press End to exit the measurement.

Step 19: Press Save to store data.

Step 20: To save the method, press the main menu [Method] | [Save Current Method] to save.

Step 21: Print the results: press Print to enter print preview.

Description of graphite furnace measurement:

- Blank:

This function activates a blank reading; the system will stop after the first solution (the blank) has been determined. The blank value determined will be automatically subtracted from the samples measured later.

- Start:

Run a complete determination with blank, standards and samples as defined in the relevant page (see above).

- Auto Zero:

sets the current absorbance to zero

- ReSlope:

Recalculate the slope of the calibration curve by using a pre-defined standard solution. While the determinations are in progress, the “Measure page is filled with results:

- Abs:

Numerical value of the corrected absorbance of the analyze- element after background correction. Depending on the selected measurement mode peak absorbance or integrated absorbance. Integrated absorbance is strongly recommended for graphite furnace determinations

- Total:

the numerical value of the total absorbance determined by the hollow cathode lamp.

- Try:

This function runs through a complete furnace cycle without sample introduction and data collection. It is used to clean the graphite tube or to heat out the furnace after an erroneous introduction of liquid.

- BG:

the background absorbance determined by the D2 lamp or during the high pulse phase, if Smith-Hieftje background correction is applied.

- Burn Out:

quick heating form 1000 degree to 2650 degree to clean the furnace

- Graph:

the time resolved graphics of the atomization pulse can be displayed and stored separately if the selection is made.

Note:

1. Before starting an analysis, activation of the ‘try’ function is recommended to run clean out.

2. The analysis can always be terminated with the button “end” in case of improper operation or other problems.

3. The maximum allowed temperature input in the “heating parameters page” is 2700 °C. If the value is higher the software will automatically reduce it to 2700 °C. The recommend maximum temperature setting is 2650°C for 6 s. It is strongly advised against setting higher temperatures and hold times. This would reduce the lifetime of the graphite tubes substantially and/or severely compromise the analytical quality of the measurement.

4. When the determinations are finished, click the ‘End’ or the cross icon close the window. When the automatic run is finished, the system will stop. You can always terminate by clicking the “end” button.

3.5 Graphite furnace autosampler: ellipsis

3.6 Self absorption background correction measurement

3.6.1 Summary:

The self absorption calibration uses the broadening of the self absorption line of the hollow cathode lamp under strong current to measure the background. It can be used for full band correction with sufficient light energy, It is helpful to improve the signal-to-noise ratio and background correction performance (the above two points are better than those of tritium gas lamp and Zeeman), and there is no need to set other beam combiners or polarizing elements in the optical path. It is a simple and feasible background correction method. The sensitivity of self-priming correction mainly depends on the self-priming degree of hollow cathode lamp under the action of emphasized pulse current. When the pulse current is strong, the element The more serious the self absorption of (cadmium, lead, cobalt, antimony, manganese, silver, nickel, tellurium, zinc, etc.), the smaller the sensitivity loss. If the self absorption of some elements (aluminum, vanadium, barium, molybdenum, platinum, palladium, tungsten, titanium, silicon, calcium, strontium, germanium, etc.) is not obvious, it indicates that it is not easy to use self absorption correction, otherwise the sensitivity will be greatly reduced.

The steps of self absorption background correction measurement are basically the same as those of deuterium lamp background correction and no background correction measurement. The only difference is that self absorption background correction will automatically optimize the background correction.

3.6.2 **Testing step**

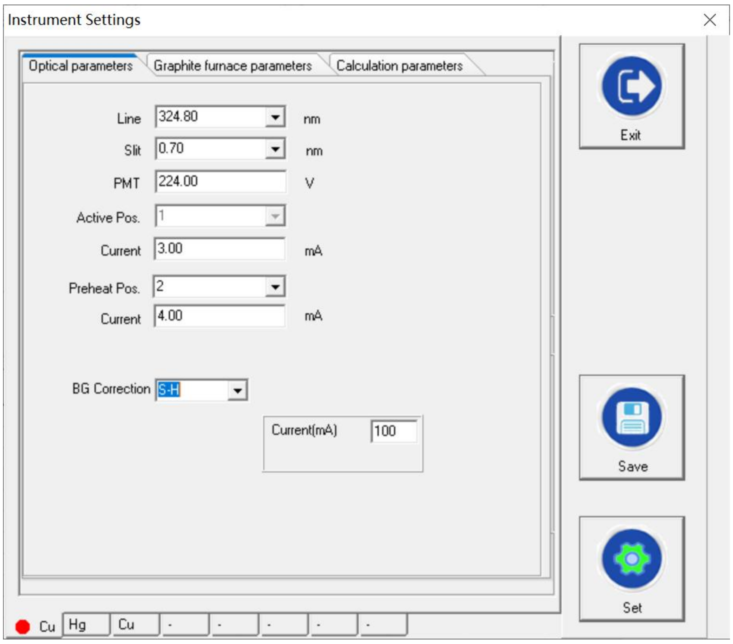
Step 1: sample setting.

Step 2: select the element.

Step 3: instrument condition setting

Select "self absorption background correction" for "background correction"; The "peak current " is 0 – 400 mA.

S-H background correction requires the input of a boost current. The default value, recommended as well in the cookbook, should be used.



Line 324.80 nm

Slit 0.70 nm

PMT 224.00 V

Active Pos. 1

Current 3.00 mA

Preheat Pos. 2

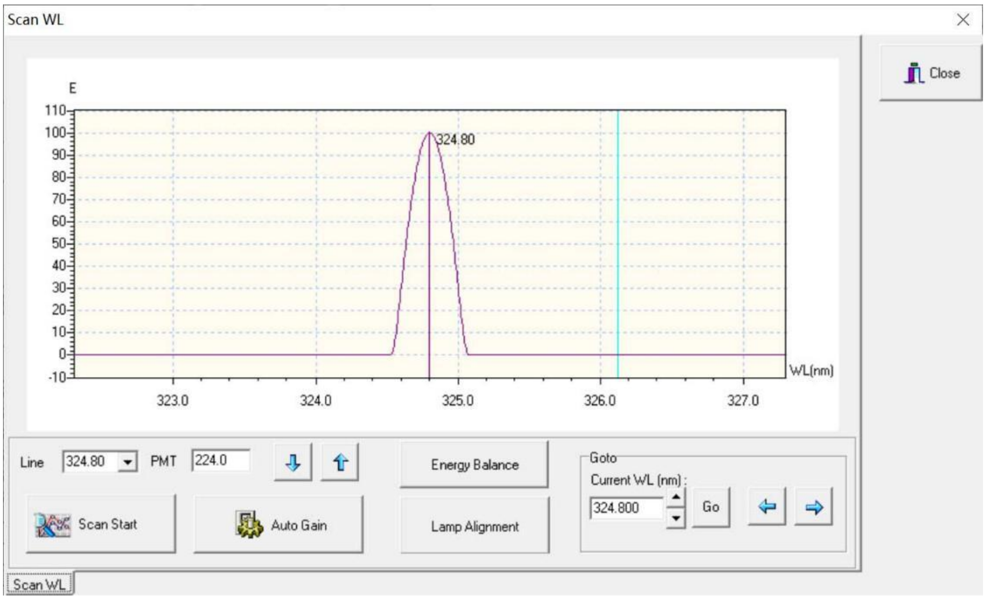
Current 4.00 mA

BG Correction S-H

Current(mA) 100

Step 4: "Wavelength peak seeking":

Click "set condition" to enter the wavelength peak seeking interface, and conduct peak seeking and adjust energy; Or click the "automatic peak seeking" button to automatically find the wavelength of the analysis line. After scanning, you can use the "automatic energy adjustment" button to automatically adjust the high voltage; If the negative high voltage is high (the energy is low), the position of the lamp can be adjusted according to "automatic fine adjustment of lamp position" and "longitudinal fine adjustment of lamp position" to optimize the energy.



Line 324.80

PMT 224.0

Current WL (nm): 324.800

Step 5: Exit "wavelength peak seeking", the software will automatically enter the background correction optimization interface, and click OK to exit.

Step 6: Auto-zero: click Zero.

Step 7: Sample injection, click Measure to enter the measurement window for measurement. The current sample number is displayed in the window title, and press Start key to make a single measurement.

Step 8: Press end to exit the measurement.

Step 9: Press Save to store data.

Step 10: To save the method, press [method] | [save current method] in the main menu to save it.

Step 11: Print results. Press Print to enter print preview

3.7 Flame Emission Technique

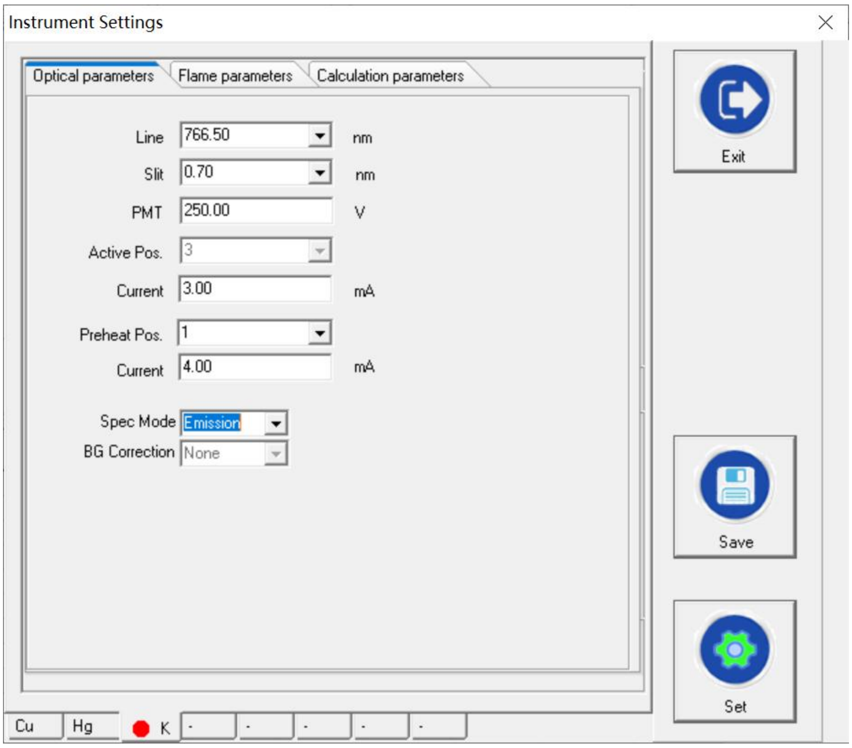
Atomic emission spectroscopy (AES) is a method that uses the intensity of light emitted from a flame at a wavelength to determine the quantity of an element in a sample. The wavelength of the atomic spectral line determines the identity of the element while the intensity of the emitted light is proportional to the number of atoms of the element. The heat from the flame evaporates the solvent and breaks chemical bonds to create free atoms. The thermal energy also excites the atoms into excited electronic states that subsequently emit light when they return to the ground electronic state. Each element emits light at a characteristic wavelength, which is dispersed by a grating or prism and detected in the spectrometer. A frequent application of the emission measurement with the flame is e.g. the control of electrolytes in body fluids.

Step:

Step 1: sample setting.

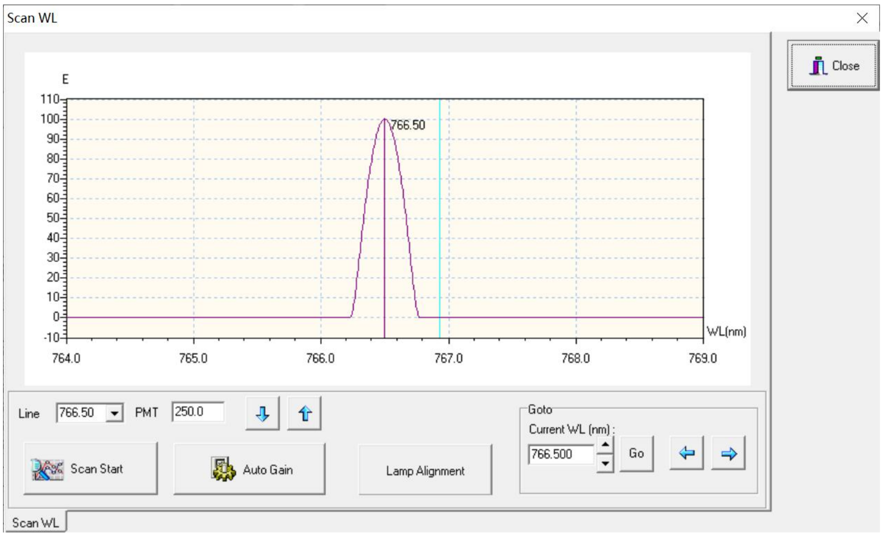
Step 2: select the element.

Step 3: instrument condition setting: select atomic emission spectrometry in the instrument condition setting interface, as shown in the figure below

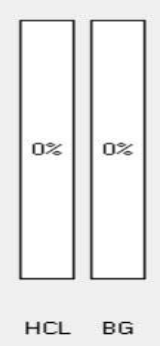


Line 766.50 nm
Slit 0.70 nm
PMT 250.00 V
Active Pos. 3
Current 3.00 mA
Preheat Pos. 1
Current 4.00 mA
Spec Mode Emission
BG Correction None

Step 4: Select Parameter set to enter the wavelength scan.



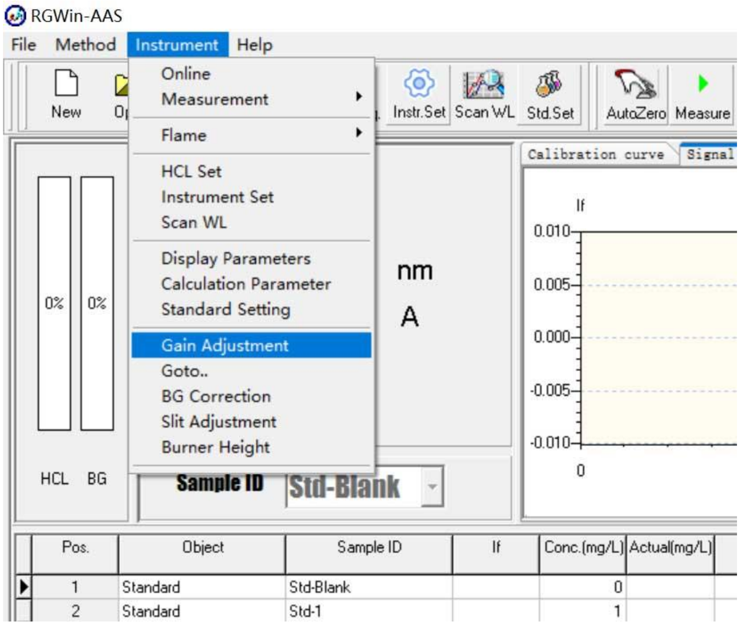
Step 5: Close the current window to exit the wavelength scan. The energy level on the energy bar on the main interface will reduce to zero due to the switch off the hollow-cathode lamp.



Step 6: Set up the standard solution parameters.

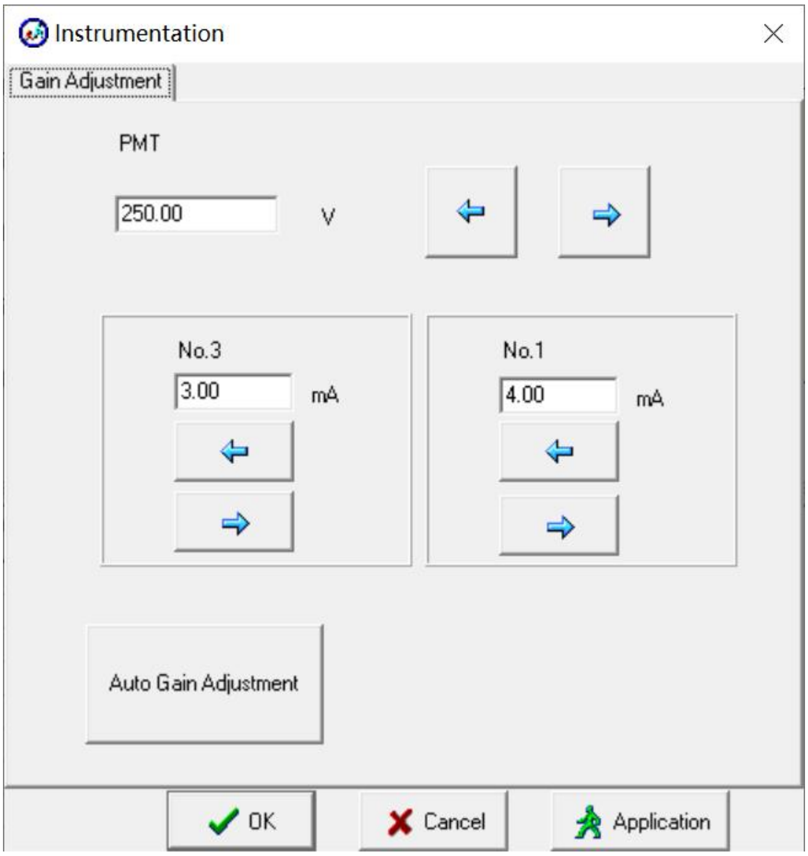
Step 7. After the parameter is set, switch on the air compressor and ethyne cylinder and ignite the flame.

Step 8. Aspirate the standard solution with the highest concentration in the series, adjust the PMT voltage to set the emission intensity to a range between 2-3 arbitrary intensity units



Sample ID: Std-Blank

Pos.	Object	Sample ID	If	Conc.(mg/L)	Actual(mg/L)
1	Standard	Std-Blank		0	
2	Standard	Std-1		1	



PMT 250.00 V

No.3 3.00 mA

No.1 4.00 mA

Step 9. After the emission intensity is adjusted, use blank solution to zero the reading. Then measure each standard solution respectively. Measure the sample afterwards.

Step 10: Click "end" to exit the measurement.

Step 11: Click "Save" to save data.

Step 12: To save the method, press [method] | [save current method] in the main menu to save it.

Step 13: Print results. Click "Print" to enter print preview

Note :

- Select air-ethyne flame or nitrous oxide-ethyne flame according to the analytical requirement.
- Use analytical grade solvents to avoid blank problems.
- Use glass or plastic volumetric flasks to prepare standard solutions.
- The burner head and mixing chamber should be thoroughly cleaned to prevent contamination.

Chapter 4 Maintenance and Trouble Shooting

Detailed information of setting up a procedure are given in chapters 4 and 5. In this section additional important information is provided to ensure best long-term performance of spectrometer and accessories.

- Make sure that the furnace is aligned properly in the light beam. Obstructing the beam by the furnace may prevent the system from performing an accurate wavelength and detector set-up routine.

- Make sure that the hollow cathode lamp operates at the recommended lamp current. Currents significantly higher than the recommended values may result in lower sensitivity (higher characteristic mass) and shorter lifetime.

- Use the recommended spectral bandwidth.

For single analytical lines, a wider slit width can lower the noise. Lower slit widths will generally result in increasing noise. The recommended value usually provides the best signal/noise compromise.

For a lamp profile showing several lines in close vicinity, a lower slit width is recommended to separate resonant from non-resonant lines. A wider slit width might reduce noise but at the cost of a strongly reduced sensitivity and a bent calibration curve at low concentrations.

■ Initial Setup

The AAS are equipped with a computer-controlled burner height adjustment. The horizontal position of the burner in the light beam is adjusted to its optimal position by the service engineer during installation or maintenance. Note that optimal sensitivity is only obtained when the burner rotation is set to 0°, i.e. the burner head is turned to its standard dead stop.

The burner head will vignette a small portion of the light beam if moved to its max. possible height (5mm). The best compromise between sensitivity and an unrestricted light path is obtained at a burner height of approximately 7 mm. This can be checked by following the light path of a copper hollow-cathode lamp with a piece of white paper. Move the burner vertically until the burner head commences to block the light beam. Memorize this position. From there, the optimal absorbance can be fine-tuned using a standard solution, which will provide an absorbance between 0.1A and 0.4A.

■ Maintenance and service

Atomic Absorption Spectrometers are highly developed systems which require regular maintenance. For Furnace AAS, all graphite parts, tubes and contact cylinders are the main areas for maintenance besides the spectrometer. The auto sampler tubing requires regular checks. The gas supply including all tubing must be clean, dry and immaculate. Regular maintenance will assure security, improve the quality of the analytical result, increase the lifetime of the equipment and reduce the probability of instrument downtime.

➢ General Instrument

It is recommended that the instrument is cleaned daily to avoid accumulating dust and condensed vapors. If contamination is observed on lamp and/or atomizer windows, these should be wiped off with optical tissue moistened with ethanol. Spilled liquids such as acidified solutions or organic solvents should be removed from instrument and accessories immediately using water or a mild detergent solution.

The instrument should not be exposed to corrosive vapors or a dusty environment. If the conditions are unavoidable, a regular service on the optical system should be scheduled to maintain the quality of the optical system. In general, an annual service of the instrument and its accessories by an authorized Spectrum Instruments´ engineer is strongly recommended.

- Gas supply and Ventilation
- Flame Gas supply and Ventilation

Three gases are used in the flame atomic absorption spectrophotometer, including ethyne as fuel gas and compressed air or nitrous oxide as an oxidant. Since potentially harmful gases may be produced during operation, enough ventilation is required with a capacity of about 10m³/min. The supply pipework system used for the instrument requires a regular check for leaks, especially after changing the gas steel bottle. Interior tubing should not be ignored, the polymer tubes inside the instrument should be changed immediately when they are appeared to be aged or frayed. This requires regular service by an authorized engineer.

It is crucial to ensure enough ventilation to protect against harmful gases. The importance of gas supplies and ventilation have been discussed earlier. The proper function and safety of valves and tubing must be checked on a regular basis by authorized personnel. This will also apply to the specified function of the ventilation.

An air compressor with pressure reservoirs is offered for Spectrum flame AAS equipment. Other sources for pressurized air, which can provide a stable pressure of 750kPa and a constant flow of > 10 L/min of air can be used as well. However, dust and moisture filters must be installed. Improper gas supply may also cause excessive noise during analysis. The filters must be checked, cleaned, and replaced regularly. When using an air compressor, drain water daily.

- Furnace Gas Supply And Ventilation.

Note: Moisture by the gas supplies will inevitably damage the automatic gas boxes! Two gases are used in furnace atomic absorption spectrophotometer, including argon as protective and purge gas and water as coolant. Because potential harmful gases may be produced during operation, enough ventilation is required with the capacity of about 10m³/min. Piped supplies used for the instrument requires a regular check for leaks, especially after changing the gas cylinder. The interior tubing should not be ignored, the polymer tubes inside the instrument should be checked on a regular basis by Instrument company´ authorized service. Changing of interior tubing shall only be processed by authorized engineers.

The importance of gas supplies and ventilation have been discussed earlier. The proper function and safety of valves and tubing must be checked on a regular basis by authorized personnel. This holds true for the specified function of the ventilation as well. Notes a cooling system is offered for Spectrum Furnace AAS equipment. Other sources should meet the specification.

- Flame Atomizer

Routine maintenance is required for the nebulizer, mixing chamber and burner.

- Nebulizer

Clean the nebulizer at the end of each working shift for about 5 minutes with 2% acid and rinse it with distilled water for about five minutes. Check that the capillary is correctly fixed to the needle of the nebulizer. Make sure that the nebulizer capillary remains its approximate length of 300 mm for manual sample introduction and 1000 mm for operation with an auto-sampler. Check for corrosion on the impact bead occasionally. The integrity of the impactor may have significant influence on the sensitivity and reproducibility of the analytical signal. Make sure that the impact bead is installed correctly. Detailed maintenance procedures of the nebulizer can be found in the appendix.

➢ Mixing Chamber

The mixing chamber should be dismantled and cleaned on a regular basis. The frequency of cleaning depends on the type of samples analyzed. Clean the chamber parts thoroughly with a mild aqueous detergent solution, rinse in distilled water afterwards and dry. Reassemble the chamber, checking the connections for tightness and blockage and refill the liquid siphon through the holes in the liquid siphon. Make sure that the drain hose is completely discharged into the waste bottle. Frequently dispose of the waste.

➢ Furnace Atomizer

Routine maintenance is required for the quartz window, contact.

➢ Graphite Electrodes

At frequent intervals and inspect the inside of the graphite electrodes, which are mounted in the center cooling block. Also check the contact surfaces of the electrodes.

Using a cotton bud or similar clean the graphite surfaces to remove any loose graphite or other material that may be present. Check to ensure that the bore and the injection hole are free of loose carbon or sample residues.

Check for any pitting on the electrode surface. If necessary, replace the electrodes.

➢ Hoses and Connections

Regularly inspect all hoses and connections (gas and water). You should immediately contact spectrum instrument service engineer if you find any leaks.

➢ Cleaning Quartz Windows

Quartz window assemblies on both side of the furnace need to be cleaned regularly. To remove a window assembly, carefully clean both sides of the quartz windows. Use only alcohol and an optical tissue. Never use coarse cloths or abrasive cleaning agents. Inspect the inside of the window mounting and clean off any contamination.

While the window is removed, check the O-ring-seal on the base of the assembly. If these are damaged, they should be replaced. If necessary, the complete window assembly can be placed in a detergent solution in an ultrasonic bath. The sonication may help to remove stubborn deposits from the window. After sonication, rinse the window with distilled water and allow to dry before refitting. To replace the window assemblies, push and twist the assembly back into the electrode housing.

- Removing and Replacing Electrodes

When you first set up the graphite furnace, the electrodes that connect to the graphite tube will appear clean, shiny and smooth. However, the electrodes may become pitted or eroded, and you may see a drop-in precision after used for a period. You should then replace the electrodes. A tool is provided, which enables you to remove and replace the electrodes. A regular visual check of the electrodes is recommended every time when changing tubes. A thorough inspection after each 10 graphite tube replacements is strongly recommended.

Chapter 5 :Troubleshooting

Instrument

This section contains answers to problems you may encounter during the installation/use of the instrument.

Flame Parts

Low Absorbance

If the absorbance is lower than normal (characteristic concentration more than 30% above the cookbook value), one of the following situations might be the cause:

1. Sample uptake rate of nebulizer too low: low sample uptake rate usually results in lower absorbance. The uptake rate can be measured by aspirating distilled water from a graduated cylinder for a minute. It should be in the range of about 5 mL/min. Low sample uptake rate below 4 mL/min may be caused by blockage or leakage. Nebulizers are subject to wear and may have to be replaced after several months of continuous operation.
2. If the nebulizer is not aspirating at all, it may be blocked. This situation can occur after aspirating solutions with highly dissolved solids, e.g. seawater or clinical samples. In this case, remove the tubing for oxidant and sampling and carefully remove the nebulizer from the mixing chamber. Remove the impact bead. Use one of the cleaning wires provided with the nebulizer to clean the capillary from the rear side. However, the nebulizer should never be dismantled. After successful cleaning, introduce the nebulizer into the mixing chamber in reverse order. Aspirate distilled water for 5 minutes to thoroughly clean the nebulizer and determine the suction rate.
3. To reduce the risk of blockage, avoid prolonged aspiration of samples containing high concentrations of dissolved solids for longer than 20 seconds and aspirate blank alternately with the sample. The sampling capillary tube may occasionally be blocked by particles which may dramatically reduce the sample uptake rate. Replace the capillary tube if the

blockage occurs. Using the Flame Micro Injection Device (FMID) is strongly recommended if samples with high concentrations of dissolved solids and require to be analyzed. FMID assures that short sampling periods are followed by an automated flushing procedure. FMID is available as an accessory from Spectrum Instruments Aust PTY LTD.

4. Do not attempt to disassemble the metal nebulizer capillary from its body. Defragmentation is very delicate and should usually be done by trained personnel or experience users only. A video instruction is available upon request, showing the defragmentation of the nebulizer.
5. Whenever the nebulizer is removed from the mixing chamber and mounted again, make sure that all fittings and screws are connected tightly. Improper re-installation might result in leakages between the mixing chamber, nebulizer and nebulizer gas (oxidant). Checking for leaks can be executed by soap solution.

   The glass atomizer is fragile, so it is strictly prohibited to take it out of the atomization chamber without trained personnel!

6. Check the fuel and oxidant gas flow rate: The composition and stoichiometry of the flame can affect the characteristic concentration of the analyze element under investigation. Elements like barium, molybdenum and silicon require a fuel-rich flame to create a reducing environment, while elements like cadmium, copper and nickel are more easily atomized in an oxidizing environment, i.e. in a fuel-lean flame. When adjusting the flame conditions, it is recommended to adjust the flow of fuel gas and keep the oxidant flow to gain maximum absorbance while aspirating a standard solution. The oxidant flow can also be optimized in a similar way.
7. Flame atomic absorption spectrometry is the trace analytical technique with the fewest interferences caused by matrix. Yet, the composition, particles and type of solvent may influence the characteristic concentration with improperly calibrate cause interferences. Such interferences are classified into physical, chemical and ionization interferences, all of them are described in literature. The recommended conditions are optimal standard acidified

solutions. They can be optimized, if complex matrix is present in the solutions for measurement. In these cases, an optimization of the recommended conditions often result in better analytical quality.

8. As described earlier, the process of atomic absorption strongly depends on the quality (line width) of the emitted element profile. Worn out line sources (HC-lamps) may result in a significant reduction in sensitivity. In some cases, the lamps will still provide enough energy, But the emission may be broadband, no longer suited for atomic absorption. A typical lamp lifetime is about5000 mA*h. Make sure to check the line source with respect to current and hours of operation.

- Excessive Noise Or Unstable Signals

The best possible reproducibility will be obtained if the spectrometer is operated under the recommended conditions without ignited flame.

It is recommended to generate a reference set of data under the recommended conditions based on at least 10 replicates of the photometric noise, i.e. the spectrometer without flame.

If excessive noise is experienced during an analytical measurement, the performance of the spectrometer can always be benchmarked using these reference data

Possible sources of excessive baseline noise may be:

5. Instrument conditions change during warm-up. Hollow-cathode lamps require several minutes until the light flux and the width of the emission profile are stable. It is recommended to start the measurements 5 minutes after lamp ignition or with a pre-warmed lamp. Warm-up will also slightly influence the performance of burner heads. It is recommended to keep the flame running for 1-2 minutes before starting the analytical measurements.
6. Inadequately aligned line source Use the automatic lamp position adjustment function of the software to align the lamp, to achieve maximum light throughput.
7. Slight displacement of wavelength Some elements, such as cobalt, iron or nickel, have complex spectra. Peaking these elements may require extra care.

4. Cleanliness of windows and mirrors All lenses, mirrors and windows must be kept clean. To clean

the lamp windows and sample compartment windows, a soft optical tissue moistened with ethanol or isopropanol should be used. Careful not to scratch the surfaces. Let the surface dry in the air. The optical path and monochromator are sealed and protected from dust in the surrounding environment. Fine dust and laboratory fumes will nevertheless gradually accumulate on the mirrors and lenses inside the sealed parts. While it is not recommended for the users to open the sealed parts, a regular service will help to keep up the performance of the instrument´s optical system.

5. Type of element. The elements exhibit different noise levels, depending on the influence on the excitation process in the lamp and on the required slit width.

6. Flame absorbance at short wavelengths Analytical flames absorb light. The magnitude of absorption is dependent on the wavelength and type of flame. The baseline noise of elements determined at short wavelengths (e.g. Arsenic, Zinc) and or nitrous oxide / ethyne flames (e.g. Phosphorus) is usually higher than for elements determined at longer wavelengths. Minimizing the noise may still be possible under these conditions:

Background correction will help reduce the noise introduced by flame flicker in these cases. Use the Smith-Hieftje background corrector may additionally reduce noise.

The burner height relative to the light beam may be optimized, such that an optimal compromise between lowest possible noise and adequate sensitivity may be found. Always start optimization from the recommended values.

Prevent disturbance of the flame by draft. Always keep windows and doors closed in the laboratory. Keep the safety door to the burner compartment closed while operating the flame.

7. Strong atomic emission will occur if high concentrations of certain elements, including e.g. sodium, potassium, calcium, strontium, barium and others are present in the matrix. These will burden the instrument’s emission correction and may result in higher noise. If possible, increase the lamp current to produce a higher source intensity.

Furnace Parts

Low Absorbance

If absorbance is lower than normal (characteristic mass concentration more than 30% higher than the cookbook value), one of the following situations might be the reason:

1. Check and optimize the heating parameter.
2. Change to a new lamp and use lower lamp current.
3. Change to a new tube.
4. Make sure that the concentration of the reference solution is accurate.

Possible sources of excessive baseline noise may be:

1. Instrument conditions change during warm-up.

   Hollow cathode lamps require several minutes until the light flux output and the width of the emission profile are stable. It is recommended to start the determinations 5 minutes after lamp ignition or with a pre-warmed lamp.

2. Inadequately aligned line source

   Use the automatic lamp position adjustment function of the software to align the lamp, in order to achieve maximum light throughput.

3. Slight displacement of wavelength.

   Some elements, such as cobalt, iron or nickel have complex spectra. Peaking these elements may require extra care.

4. Cleanliness of windows and mirrors.

   All lenses, mirrors and windows must be kept clean. To clean the lamp windows and sample compartment windows, a soft optical tissue moistened with ethanol or iso-propanol should be used. Make sure not to injure the surfaces with scratches. Let the surface dry in the air. The optical path and the monochromator are sealed and protected from dust of the surrounding environment. Fine dust and laboratory fumes will nevertheless gradually accumulate on the mirrors and lenses inside the sealed parts. While it is not recommended for the users to open the sealed parts, a regular service will help to keep up the performance of the instrument´s optical system.

5. Type of element.

   The elements exhibit different noise levels which depend on the influence on the excitation process in the lamp and on the required slit width

The software is not communicating with the instrument.

- Ensure the computer has been rebooted after installing the software.
- Check all cables and connections.

- Check that the instrument and all associated accessories are turned on.
- Ensure that the computer comport matches the setting required by the software.

➢ After peaking, the emission profile shows a straight or irregular line without characteristic peak, or a high PMT voltage:

1. Observation :The hollow cathode lamp is not glowing ;

   Solution :Properly insert the lamp, set the condition and peak again

2. Observation : Lamp position or element is not chosen correctly;

   Solution : Choose the right lamp and element

3. Observation : Lamp cannot be lit

   Solution : Check if lamp is defective by installing it into

   another position in the lamp turret. Replace lamp if it does not light up.

4. Observation : Atomizer blocks the optical path;

   Solution : Set the atomizer to proper position

5. Observation : High PMT-voltage (~500V);

   Solution : Lamp defective or dirty windows or atomizer blocking the optical path.

➢ Unexpected spectra

1. Observation : Flat peak, energy at 100% ;

   Possible source of error: Energy overflow

   Solution : Click auto balance energy in the software. Peak again until a sharp peak appears.

2. Observation : Noisy graph without explicit peak;

   Possible source of error: Element selection incorrect. Wavelength deviation is beyond the peaking range

   Solution : Use correct lamp or set the correct element. If the peak is beyond the range, Service required.

➢ Unable to light up the Hollow Cathode Lamp

1. Possible source of error: Lamp current error (0mA)

   Solution : Reset lamp current

2. Possible source of error: Software indicates “instrument off- line”

   Solution : Refer “The software is not communicating with the instrument”

3. Possible source of error: Another lamp in the same position can be lit

   Solution : Lamp defective

4. Possible source of error: Another lamp in the same position is not operative as well

   Solution : Lamp board defective. Service required.

- D2 Lamps fault

1. Observation : Deuterium lamp flickers ;

   Possible source of error: 1, Defective lamp or life span reached 2, defective electrical connection

   Solution : 1, Replace the lamp 2, Check connection of cable.

2. Observation : Deuterium lamp cannot be lit;

   Possible source of error: 1, Defective lamp or life span reached 2, defective electrical connection 3, Lamp board defect

   Solution : 1, Replace the lamp 2, Check and reconnect 3, Service required.

Thank you for reading this manual carefully. We hope the contents in this manual will help you use the instrument correctly. If you have any questions or difficulties during the use of the instrument, you can contact us freely. Our after-sales team will help you solve your problems in time and quickly.