AA320N ATOMIC ABSORPTION SPECTROPHOTOMETER

User Manual



Please read operating manual before installation and operation.

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1. General Description

1.1 Main Applications

Model AA320N Atomic Absorption Spectrophotometer can be used to measure constant quantity and trace metal elements in various samples through atomic absorption analysis and flame emission analysis by flame method, graphite furnace method and hydride method.

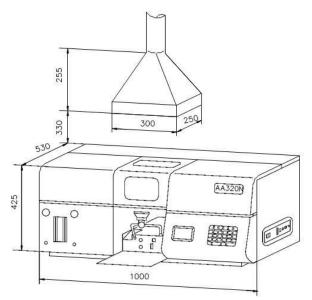
1.2 Explanation of Model

- AA —— Atomic absorption
- 3 —— Product of atomic absorption series.
- 20 —— Development order number
- N —— Modified order number

1.3 Environment Conditions

- 1) Ambient Temperature: $10^{\circ}C 30^{\circ}C$
- 2) Indoor relative humidity shall not be greater than 85%.
- 3) No vibration and magnetic field interference
- 4) No corrosive gases in the room
- 5) Power: 220V±22V, 3A, 50Hz±1Hz

2. Construction Characteristics and Working Principle



2.1 Appearance and Ventilation Device



2.2 Working Principle

Atomic absorption spectrophotometer is a measurement method following the principle of light absorption by ground state atom at a characteristic wavelength. Normally, a hollow cathode lamp is used as a light source to emit the spectrum with a given element's characteristic wavelength. When such a light-bean passes through a sample containing a ground state atom, part of light intensity will be absorbed. The absorption depends on the atomic concentration. The atomic concentration of a sample can thus be worked out according to the light absorption.

When a light beam with a light intensity of I_0 passes a medium whose atomic concentration of element to be tested is C, the light intensity will be weakened to I, which complies with the

Law of Brown-Bill.

Where:

A Absorptance

Io In-coming light intensity

I Out-going light intensity

K Absorption Coefficient

C Atomic concentration of element

L Distance traveled by a light beam through the sample

The concentration of the sample to be measured can be worked out by means of this relationship.

3. Technical Characteristics

3.1 Main Parameters

Wavelength range	190.0nm ~ 900.0nm		
Wavelength accuracy	±0.5nm		
Wavelength repeatability	≤0.3nm		
Wavelength scanning speed	1.2nm/min; 300nm/min		
Resolution	< 40%		
Characteristic concentration	≤ 0.04µg/ml/1% (Copper)		
Detection limit	≤ 0.008µg/ml (Copper)		

4. Dimensions and Weight

4.1 Dimensions

1000mm×530mm×425mm

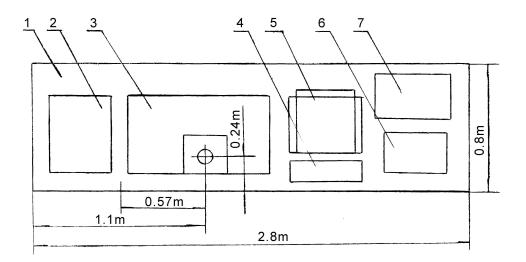
4.2 Weight

130kg

5. Installation and Commissioning

5.1 Test Table

The instrument shall be put on the flat top of table of firm and stable structure. The width of the table shall be greater than 0.8m and the length shall be good enough to hold the main instrument and accessories (refer to Fig. 5.1). If a water discharge pipe is not to be exposed, a hole of 70mm diameter shall be taped at a proper position on the top of the table for the pipe. For the convenience of repair and maintenance, a passage of 0.5m width shall be reserved behind the table.



Test table 2. Graphite furnace power supply (option) 3. Spectrophotomer
 Keyboard 5. Computer 6. Printer 7. Desk-top recorder

Figure 5.1 Reference Dimensions for Placing Test Table and Instrument

5.2 Power Supply and Gas Supply

If a graphite furnace system is used, power shall be supplied separately from the power supplied to the main instrument due to its large power load.

An air compressor shall be placed several meters away from the main instrument. There shall be good ventilation and the connecting hose shall not be close to the heat source. Acetylene cylinder-with an anti-flashback device shall be prepared by users. Cylinders must be placed in a space with good ventilation. Smoke and fire are strictly prohibited within 5 meters to avoid explosion.

5.3 Main Instrument

5.3.1

a) After the instrument is placed on a stable test table, a ventilation device must be mounted on the combustion chamber to exhaust in-door's waste gases.

b) The premix chamber and burner shall be fixed on the base seat of the combustion chamber.

A 10mm hose shall be installed with one end on the waste solution outlet of the premix chamber and another end going through the base plate to insert in the waste solution container, making the hose in a loop of 100mm diameter. Water shall be added into the premix chamber; so that the loop will be full of water to form waterseal (refer to Fig. 5.3.1).

- 1. Burner
- 2. Premix chamber
- 3. Atomizer
- 4. Sample-in capillary tube
- 5. Base plate of combustion chamber
- 6. Waste solution pipe
- 7. Base plate of instrument
- 8. Test table plate
- 9. Tying stripe
- 10. Water seal loop
- 11. Waste solution container
- 12. Waste solution

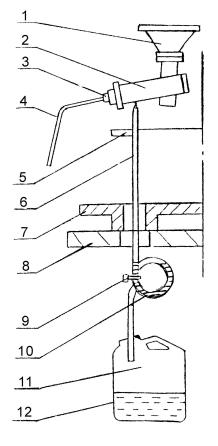
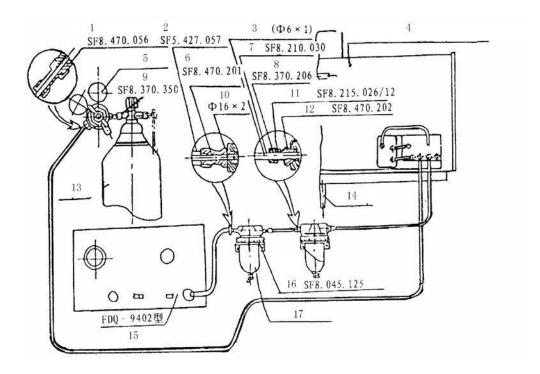


Figure 5.3.1 Waste Solution Discharge System in the Premix Chamber

c) The Atomizer shall be installed in the end-cover in the front of the premix chamber. After being tightened, a capillary tube shall be inserted to connect combustion-supporting gas tube with atomizer and mixed-gas (acetylene) tube with the premix chamber respectively.
d) The gas path connector is located at the back of the instrument, refer to Fig.5.3.2 and Fig5.3.3.



1. Connector2. Conduit Parts3. PU Tube4. AA320NDorsal View5. Acetylene Pressure-reducing Valve6. Input Connector7. Bush

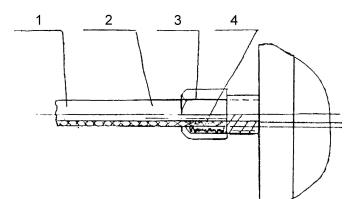
8. Seal Ring 9. Seal Washer 10. O-ring 11. Nipple

12. Output Connector 13. Acetylene Cylinder

14. Waste Solution Pipe 15. Oil-free Air Compressor 16. Seat

17. Water Knockout Gas Filter

Figure 5.3.2 Schematic Diagram of Gas Path Connection



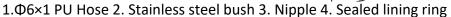
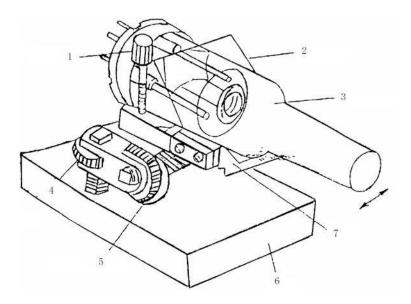


Figure 5.3.3 Schematic Diagram of Connector Installation

e) Light-source chamber is on the upper-left of the instrument. Lamp pins shall be inserted to the lamp power socket. Lift the torsional spring and place the lamp into the lamp holder (Fig.5.3.4).



1. Adjustment Screw 2. Torsional Spring 3. Hollow cathode lamp

4. High-low Adjustment Drum 5. Right-left Movement Drum 6. Base Plate

7. Lamp Holder

Figure 5.3.4 Installation of Lamp Holder

f) Insert the power line of the instrument, the cable and power line of the printer into their

corresponding sockets respectively.

5.3.3 for gas path connection and connectors' installation.

1) A hose of $\phi 6 \times 1$ shall be installed with one end connecting to the air inlet of the instrument

while the other end to the air compressor.

2) A hose of $\phi 6 \times 1$ shall be installed with one end connecting to the acetylene inlet while the other end to the acetylene cylinder.

3) A hose of $\phi 6 \times 1$ shall be used to connect end to end of three connectors marked with

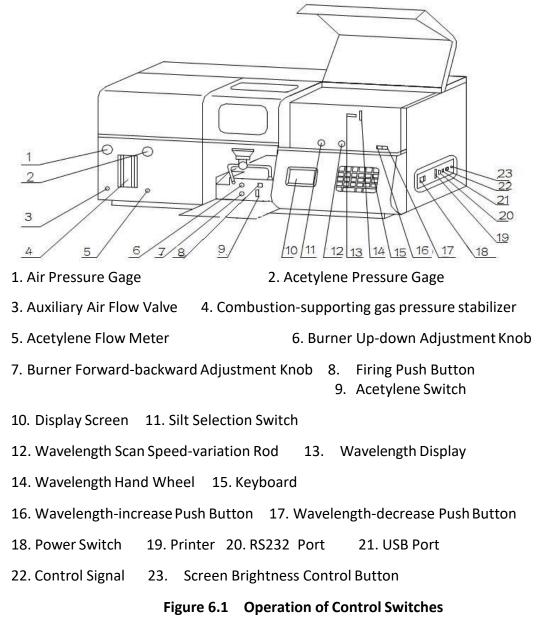
"Atomization", "Combustion Gas" and "Firing Acetylene" respectively.

NOTE :

Do not use a copper tube whose copper contents are greater than 65% to connect acetylene gas path .

6. Basic Operation

6.1 Description of Operation Switches



6.2 Operation

6.2.1 Adjustment of Element Lamp

1) Open the upper-left door on the instrument, insert the hollow cathode lamp into the lamp power cable seat and pressed by spring when it is in the lamp holder and align the lamp to the marks on the holder.

2) Turn on the lamp power toggle switch nearby the lamp holder and move the cursor to I (Lamp Current) to set a suitable lamp current in F2 page.

3) Open two pieces of light barriers on the right wall of combustion chamber to let the light beam pass through the combustion chamber.

4) Select a suitable wavelength (If the scan speed variation rod ispulled out, the scan speed is 300nm/min; pushed in, the speed is 1.2nm/min; in the middle, the scanning is stopped, which can be adjustment by a hand wheel in one specified direction), slit and HV (Negative high voltage) and make the energy indicator S to about 90 in F2 page (After the hollow cathode lamp is warmed up, energy may increase).

5) Adjust wavelength with a wavelength hand wheel to make the energy indicator to the maximum value. If energy is greater than 90, adjust HV to make it return back to about 90. 6) Turn slowly adjustment wheel of hollow cathode lamp to make the energy indicator to the maximum. If energy is greater than 90, adjust HV to make it return back to about 90.

6.2.2 Beam-focusing of Burner

The path and position of a light beam of the hollow cathode lamp will exert great influence on the sensitivity of the system. Burner's slit shall be parallel to or slightly lower than the optical axis of the instrument. Operation is as follows:

1) Lower the height of burning head to be under the lightbeam.

2) Put the focus plate on the slit of the burning head and move it along the slit. Adjust the rotation handle of the burning head to make the slit be parallel to and exactly under the light beam.

3) Rotate the up-down adjustment knob of the burner to go up slowly the burner until energy just starts to change.

4) Rotate the knob another half turn counterclockwise to lower the burner further down. This may be the optimum height for a multi-element analysis, which means that the center of the light spot is on the height of 5mm in the focus plate.

5) Eject a portion of standard solution, rotate slowly the forward and back adjustment knob of the burner to reach the maximum absorption value.

6.2.3 Air-Acetylene Flame Operation

1) Check if a 100mm burning head and waste solution discharge pipe are properly installed.

2) Open the pressure-reducing valve on the acetylene cylinder until the output pressure reaches 0.07 MPa.

3) Turn on the power of air compressor to adjust the output pressure to 0.3 MPa.

4) Turn on the master power switch of the air path and combustion-supporting gas switch.

Adjust the combustion-supporting gas pressure stabilizing valve until the pressure gage indicates 0.2 MPa.

5) Rotate clockwise the auxiliary gas knob to close auxiliary gas. At that time, the indication on the flow meter only refers to the flow of atomized gas of about 5.5 L/ min. If necessary, auxiliary gas can be used but the increase of auxiliary gas will reduce the sensitivity of absorption.

6) Adjust the pressure-reducing valve on the acetylene cylinder to make the acetylene gage indication be at 0.05 MPa. Turn on the acetylene switch and adjust acetylene knob until the acetylene flow reaches about 1.5L/min.

7) Press the firing button (for about 4s) and flame from the firing nozzle will fire the burning head (if 4s later, it fails to fire, release the firing switch to avoid the firing platinum filaments being burned out. Re-fire after the acetylene flow is increased). Press the firing button first and turn the acetylene flow knob after the platinum filaments become red.

8) Adjust acetylene flow to select suitable fire foranalysis.

9) After measurement is completed, eject distilled water for several minutes first and then turn off the acetylene switch or close directly the acetylene cylinder valve to put out the fire. Lastly, turn off the combustion-supporting gas and master power switch of air path, cut off air compressor's power and release remaining gases.

> **NOTE**: To put off the fire, close acetylene first and then air!

7. Sample Measurement

7.1 F1 Page : Elements

Move the cursor to an element and press Enter key to select the element to be measured.

7.2 F2 Page : Setup

7.2.1 Description of Symbols

- 1) S: Energy of sample light beam
- 2) D_2 or R (R is invisible) : Deuterium lamp energy or reference light beam energy (when

the deuterium lamp is on, the optical door near the right side of the combustion chamber shall be closed).

- 3) HV : Negative High Voltage
- 4) I: Lamp current
- 5) WL: Wavelength
- 6) SW : Slit
- 7) PT: In the flame method, it refers the delayed time before measurement while in the

graphite furnace method, it refers to zero-adjustment time before measurement.

8) IT : Integral time, which means the measurement time. Usually it will be 2s-3s in the flame method.

9) RSP: Response time.

- 10) N: Averagetimes.
- 11) X : Time coordinate range
- 12) Y : Absorptance coordinate range

7.2.2 Parameters Setup Method

Move the cursor to the item needed. Use + or – key to select item 2,3,4,5 or 6 and use numerical key to set 3, 4,7, 8,9,10,11 or 12 item. Press Enter key to confirm after setup.

7.3 F3 Page : Condition

7.3.1 Description of Symbols

1) MODE refers to measurement Mode. AA refers to flame absorption; AE refers to flame emission; PH refers to peak height; PA refers to peak area; PH-A and PA-A refer to peak area measurement when the instrument is connected with sample auto-feeder of graphite furnace. (Operation will be done directly based on the data without the selection of operator.)

2) CURVE refers to choice of working curve. L1 refers to a linear equation passing the original point. L2 refers to a linear equation not passing the original point; NL refers to a non-linear equation ; SADD refers to the standard addition method.

3) D_2 : ON refers to the deuterium lamp is on and OFF refers the deuterium lamp is off.

4) UNIT : mg/L; μ g /L; %; μ g; ng; pg.

5) FLA refers to Flame mode. A-C refers to air-acetylene; N-C refers to nitrous oxide – acetylene.

- 6) H refers to the height of flame.
- 7) AIR refers to air flow and pressure.
- 8) C_2H_2 refers to acetylene flow and pressure.

7.3.2 Parameters Setup Method

Move the cursor to the item needed. Use Enter key to select item 1, 2, 3, 4 and 5 and use numerical key to select item 6, 7, 8 and 9 and press Enter after setting up.

7.4 F4 Page : Working Cuvre

7.4.1 Description of Symbols

- 1) S: Standard
- 2) C: Concentration
- 3) mg/L: Unit of concentration
- 4) A: Absorptance
- 5) k: Gradient
- 6) b: Intercept
- 7) r: Related coefficient

8) 0~8: 0 means that the line is for the blank solution of the standard sample, 1~8 means the number of standard samples.

7.4.2 Parameter Setup Method

C (concentration) for each line can be set up with numerical key assisted by $\leftarrow, \uparrow, \rightarrow, \downarrow, \downarrow^{\downarrow}$ key. A (absorptance) can be set up by means of automatic measurement of the instrument or keying in through the keyboard. After the completion of working curves, working points can be added, altered or deleted to re-plot the working curve. Use + and – keys to select different kinds of curves.

7.5 F5 Page : Analyses Testing

7.5.1 Description of Symbols

1) A : Absorptance 2) s : Second (unit of time)

- 3) STD0 : Standard blank
- 4) STD1 : Standard 1 and the rest may be deduced by analogy.
- 5) #0000: Sample blank (BK on the report)
- 6) #0001 : Sample 1 and the rest may be deduced by analogy.
- 7) RSLP : OFF means that gradient reset is closed. Use Enter key to select ON, the gradient reset is on.
- 8) BG : OFF means that the background curve does not displayed. Use Enter key to select On to display the background curve.
- 9) REDRAW: If a peak signal does not fall in the IT range, spectrogram can be plotted again to make it within the IT range for accurate calculation.

10) –n: Placed after the standard sample and sample number means the number of repeated testing with its range being decided by Item N in the F2 page. After the completion of n times, average absorptance and concentration of the repeated testing will be displayed.

11)Absorptance value displayed at the upper right corner AA-BG means that background is deducted.

In F5 page, the span of vertical ordinate A can be modified using \uparrow, \downarrow key or numerical keys and the span of horizontal ordinate S can be modified using \uparrow, \downarrow key or numerical keys, the maximum being 99.9s. PT and IT can be selected using $\uparrow, \downarrow, \leftarrow$ or \rightarrow key, then using + key to move right and – key to move left (the sum of the two can not be greater than value X). The number of standard sample, STD and number of sample, # can be set up using "STD" key and "#"key assisted by numerical keys.

7.5.2 Operation Method

It is assumed that there are three standards and N=2, PT=0, IT=1.5

1) Continuous Signal Flame State

First press Zero-adjustment key to make the signal be zero (if press again "-" key or change the page, the value before zero adjustment will be restored), then press Start/End key, the prompting box "STD0-1" will be appeared in the lower left corner. When spraying in the STD0 standard sample blank and pressing the numerical key after the signal is stabilized, absoptance and "Yes?" will be appeared in the upper right corner. Press Enter key to confirm. If press the Delete key, it means the value is not wanted (when a sample auto-feeder of graphite furnace is used, chose PH-A or PA-A in the Mode and the instrument will go on with the testing automatically without confirmation) and it can be redone. If "STD0-2" is appeared in the lower left corner, spraying STD0 standard sample blank and pressing the numerical key after the signal is stabilized, absorptance and "Yes?" will be appeared once again in the upper right corner. Press Enter key to display the average absorptance and concentration There will be a record in F6 page). The rest may be deduced by analogy to complete three standards and samples.

NOTE :

If the measurement of absorptance of a standarnd sample is completed, working curves' shape and related coefficients can be displayed in the F4 page. If not, the concentration of sample can not be obtained.

⁽²⁾ In the F4 page, use the Delete key to delete standard work points. To increase standard work points, input C value of concentration in F4 page and then turn

to F5 page, press standard sample key and input standard sample number with numerical key (If press the standard sample key and input 4, the prompting box of "STD4-1" will be appeared in the lower left corner). Then test the standard sample added and work curve will be automatically plotted.

(3) Repeat the test sample, in the F5 page, press # key and input the number with the numerical key (if press # key and input 9, the prompting box "#0009-1" will be appeared in the lower left corner).

2) **RSLP Operation**

The sensitivity of the instrument may vary with a slight variation of flame and atomization state. Therefore, it is necessary to check and correct the sensitivity of the instrument after a certain period of testing operation. The method is as follows:

Select at will a standard sample similar to the sample to be measured and select a time interval. After the completion of 10 samples testing, input this standard to read the absoptance and check if the absorptance is the same as before. If there is a great difference, press the standard sample key to input the corresponding sample number and turn on RSLP. Press reading key and Enter key to complete the calibration. If press the Delete key, the value will not be involved in the calibration.

3) BG Operation

When the testing is done with background deducted, move the cursor to item BG, press «key, the state ON means that it can display the time curve of background absorptance.

4) **REDRAW Operation**

Press directional keys to move upward the cursor to two small optical pillars on the axis X. Use + or - key to move small optical pillars and make the peak signal fall in the middle of two optical pillars (range between small optical pillars is the value of IT) and then move the cursor downward to REDRAW. Press Enter key, the value of peak signal will be displayed.

7.6 F6 Page : Report

7.6.1 Description of Symbols

- 1) No: Number of sample and standard sample.
- 2) C: Concentration of sample and standard sample.
- 3) mg/L : Unit of concentration
- 4) AA-BG : Sample absorption with background absorptance deducted.
- 5) BG: Background absorptance
- 6) SD : Standard deviation
- 7) RSD (%) : Relative standard deviation

In this page, data just measured can be written in automatically and sample data can be deleted with the Delete key. If press the function key and Delete key, sample data will be completely deleted but standard sample can be retained. If they need to be saved after the instrument is turned off, please use F7 function to save. In F6 page, press report key to print the analysis report of the sample in this page. The format of the report can be chosen with Yes/No in F8. If stored sample data is full, it will prompt on the screen: Data Full, which means that sample data can no longer be stored in the file and a new file shall be created.

7.7 F7 Page : Files Manage

7.7.1 Description of Symbols

- 1) Files: Name of a file
- 2) Load : Call a file
- 3) Save : Save a file
- 4) New : create a new file

The page will display the file names saved by the instrument and it can call, save, create and delete a file.

a) Call a file : Move the cursor to the file name and press 4. Then move the cursor to the item of Load and press 4 key, pages of F1, F2, F3, F4 and F6 are all become the contents of the file. b) Delete a file: Move the cursor to the file name and press Delete key. The screen will prompt Del? (Do you really want to delete?) Press4 key to delete. c) Save a file: Move the cursor to item Files and input the file name (9 places utmost) and press «key. Then move the cursor to the item Save and press «key, the file has been saved. At that time, the file name will be automatically added to the contents of F7 page (save 30 files utmost).

d) Create a new file: Move the cursor to the item New and press 4key, the screen will prompt Save? It means that is the current file saved? If saved, press 4 and all the F page is for initialization value.

7.8 F8 Page : Data Print

7.8.1 Description of Symbols

1) File Head refers to the file head of reports in F6, Yes/No --Print /Not print.

2) Standard Samples refer to the standard samples in the reports in F6 Yes/No — — Print/Not print.

3) Parameters refer to the parameters in the reports in F6. Yes/No ——Print/Notprint.

4) Item refers to the item in the report in F6. Yes/No—— Print/Not print.

5) Online refers to online print in F5. Yes/No ——Online print / Non-online print.

7.8.2 Description of Each Item

1) File Head refers to

Analytical Report

2) Standard Samples refer to the absorptance and concentration of the standard samples in

the report in F6.

3) Parameters refer to parameters set in F2 and F3.

4) Item refers to

No CONC (mg/L) AA-BG BG SD RSD (%)

5) Online refers to that data measured in F5 can be online printed after confirmation (if the printer is not turned on or is not connected, No shall be chosen, otherwise, it may stop the instrument). Move the cursor to an item, use + to choose Yes/No. If press < key, the item can

be printed. If any screen contents are needed to be printed, press Copy key.

7.9 F9 Page : Test

This page is used by the manufacture to adjust the instrument.

7.9.1 Description of Symbols

1) Baseline 1 to examine baseline stability for 30 minutes (After zero adjustment, press "Start/End" key to conduct the measurement).

2) Baseline 2 to examine the baseline stability at 10% variation of lamp current. After zero adjustment, press "Start/End" key to test the stability of 10mA. Then press "Delete" key and press + or – key to test the stability of 11mA or 9mA.) $_{\circ}$

3) Baseline 3 to examine edge energy. 4)

Resolution to examine resolution. 5)

Calculate to perform statistical measurement.

8. Selection of Optimal Conditions

8.1 Selection of Lamp Current

The large or small lamp current will affect the analytical sensitivity and precision. A smaller current can obtain a higher sensitivity but with bigger noise. Therefore, the suitable lamp current shall be carefully selected.

8.2 Selection of Flame

Suitable flame can improve the analytical sensitivity and stability. After the flame is fired, adjust the flow of acetylene to change the proportion of combustion-supporting gas and burning gas to reach different flames until the maximum absorption is obtained. When air-acetylene flame is used, small acetylene flow provides blue flame and large flow provides yellow flame. When nitrous oxide – acetylene is used, there shall be a rose inner flame of 10mm-20mm high.

8.3 Selection of Burner Position

The position of the burner, in particular, the height of it will exert great influence on the analytical sensitivity and stability. After focusing, select once again the height of the burner to search for the flame range with a maximum absorption and most stability. The position of burner shall be adjusted while the sample is absorbed under the flame conditions.

8.4 Sample Pick-up Speed

The sample pick-up speed will greatly affect the analytical sensitivity. To a certain degree, the more the amount of sample solution to be tested is sprayed into the flame, the better the absorptance is. But if too many, the absorptance will get lower.

For a metal atomizer, the sample pick-up speed can be changed by turning the adjustment nut (refer to Fig.9-1) and altering sample-in capillary tube with different caliber or length. A sample-in capillary tube with a large caliber and short length has a greater sample pick-up speed.

8.5 Utilization of a Dispersing Ball

The utilization of a dispersing ball can improve sample atomizing effect. Generally speaking, it will produce a better effect if a dispersing ball is placed near the nozzle with its center slightly lower than the axis of the atomizer.

The dispersing ball on the glass atomizer has been already fixed at the optimal position but the dispersing ball on the metal atomizer shall be fixed carefully by the operator. The installation hole of the dispersing ball is on the adjustment rod at the cover of premix chamber, which can be turned by the knob on the cover. Insert the dispersing ball into the hole on the adjustment rod and eject solution to observe atomization. After the optimal position is chosen, fix it with a small plastic screw on the adjustment rod and then close the cover of premix chamber.

8.6 Selection of Spectrum Bandwidth

Spectrum bandwidth is just referring to a slit. Under the prerequisite that non-resonance line can be separated, a wide slit shall be used as much as possible so as to improve the signal-to-noise ratio and analytical stability.

There are 6 widths of slit available in the instrument.

8.7 Selection of Wavelength

There is a table of element lamp analytical lines and burning gases in the inside of the right turnover plate of the instrument. Two working wavelengths are recommended for most element analysis. The first wavelength is the analytical line with the most or relative high sensitivity and better stability for routine analysis. The second wavelength is the analytical line with secondary or lower sensitivity for the analysis of high concentration samples.

9. Maintenance

9.1 Cleaning Burner's Slit

After firing, the whole slit shall have an evenly burning flame. In case flame is in saw-tooth form, it means that the slit needs cleaning. Put off flame first and then insert the filter paper into the slit to clean carefully. If by doing so, there is no improvement. Remove the burning head and wash it in the water with a fine and soft brush. If there are beads on it, use a piece of fine metallographic sand paper or a single face blade to scrape carefully. It is strictly prohibited to soak the slit in the acid.

9.2 Burner Cleaning

9.2.1 Ejection of Organic Samples

After ejection of organic samples (such as oils or methyl isobutyl ketone), in the following measurement, the absorptance signal may generate noise and become unstable. It is because that the organic solution pollutes the water solution sample for later measurements. In order to prevent pollution, the atomization system shall be cleaned according to the following procedures:

1) Eject blank organic sample for about 5 minutes.

2) Eject acetone for 5 minutes.

3) Lastly eject 1% HNO₃ for 5 minutes.

4) Check burner. If there are precipitates, remove the burner, clean the premix chamber and burning head with clean solution and a brush.

5) Wash waste solution discharge pipe with water. Disposal of corrosive solution shall be done according to related local regulations.

9.2.2 Ejection of High-Concentration Copper, Silver or Mercuric Salts

After ejecting high-concentration copper, silver or mercuric salts in air-acetylene flame, it may produce unstable acetylide, which may lead to explosion after it is dried. Therefore, whenever

this kind of analysis is done, one must eject pure water immediately to wash thoroughly the premix chamber, burning head and waste solution discharge pipe and check carefully if there are any residuals left.

9.3 Cleaning Atomizer and Sample-in Capillary Tube

After cleaning the burner and slit on the burning head and if the reading of absorptance is still low, it may be caused by the partial block of atomizer or sample-in capillary tube. Eject pure solvent till the following standard sample measured has a relative satisfied reading of absorptance.

NOTE :

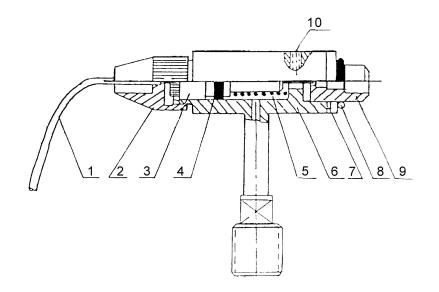
If a long sample-in capillary tube is used, ejection flow and sensitivity of sample will be reduced. In case ejection of solvent fails to eliminate the block, the sample-in capillary tube or atomizer capillary tube needs cleaning:

1. Metal Atomizer

Use soft steel wire to unclog the capillary tube to eliminate solid particles in it. If it fails, remove the atomizer for cleaning. Its structure is as shown in Fig.9-1.

2. Glass Atomizer:

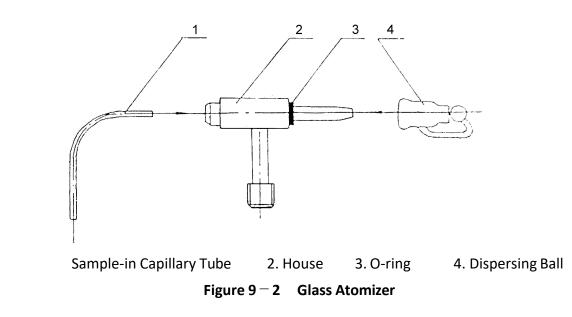
Remove the end cover from premix chamber and take out the dispersing ball and atomized gas hose from the atomizer. Blow the outlet of atomizer with atomized gas till all the solid particles are blown out. Its structure is as shown in Fig.9-2.



1. Sample-in Capillary Tube 2. Adjustment Nut3. Capillary Tube Fixing Cover4 and 8. O-ring,5. Compressing Spring6. House7. Sealing Ring

9. Nozzle 10. Adjustment Screw

Figure 9 – 1 Metal Atomizer



9.4 Gas Path Inspection

1.

If the gas path has undergone repair or dis-assembly, leakage check must be done. It is particularly so to the acetylene path. The Method is:

1 Unscrew the nipple connector of acetylene hose connected to the burner and tighten it with a accessory blind nut.

2 Close the auxiliary gas needle valve and turn on gas path electric switch,

combustion-supporting gas electric switch and acetylene electric switch. Open acetylene needle valve and stabilizer valve.

Open the valve on acetylene cylinder to reach a output pressure of 0.07MPa and then close the valve. At that time, acetylene has been sealed in the acetylene path (refer to Fig. 3-5). 4)
 Observe acetylene pressure gage for 10 minutes and the pressure drop shall not be greater than 0.01 MPa.

10. Fault Analysis

10.1 Analytical Result being Higher

Causes:

- 1) Zero point is not adjusted with a blank solvent.
- 2) Ionization inference exists.
- 3) Standard solution is polluted or improperly prepared.
- 4) Background absorption (molecular absorption or light dispersion, etc.) exists.

10.2 Analytical Result being Lower

Causes:

- 1) Chemical interference or substrate (great viscosity) interference exists.
- 2) Standard solution is improperly prepared.
- 3) Blank solution has been polluted.

10.3 Can not Reach the Specified Detection Limit

Causes:

- 1) Unsuitable expanded scale and integral time are used.
- 2) Improper flame condition or wavelength chosen leads to a very low sensitivity.
- 3) Too small lamp current affects its stability.

10.4 Can not Reach the Preset Sensitivity

Causes:

1) Analysis is done on a wrong spectrum line. Many elements have close neighboring spectrum lines, each with a different sensitivity.

- 2) Different atomizers have their different sensitivities.
- 3) Metal atomizer has a relative low sensitivity as compared to that of glass ones. 4)
- When improper flame is used, many elements are very sensitive to different flames.
- 5) Check flame conditions. Many elements are very sensitive to the proportion of burning gas

to combustion-supporting gas.

10.5 Static Noise Being Too Big

Causes:

- 1) Make sure lamp is placed in the instrument.
- 2) Two big scale expansion is selected for measurement condition.
- 3) Power voltage is too high or too low.
- 4) Lamp emission intensity is weak or electric discharge is abnormal or lamp current is too

small.

10.6 Dynamic Noise being Too Big

Causes:

1) It is caused by high absorption of flame. When testing elements in the far-ultraviolet region (such as As or Se), the analytical noise may become great.

Solution: It may sometime be improved by background calibration.

2) Lamp energy is insufficient along with the strong emission from the flame or constituents of solution. That will cause electron-multiplier phototube to produce great noise. Solutions :

a) Increase the working current of hollow cathode lamp within the maximum

current allowed.

- b) Change with a new lamp with sufficient energy.
- c) Analyze on another absorption line
- d) Eliminate main constituents of the solution generating intensified

emission through flame in a chemical way.

3) Ejection of organic sample solvent (such as oils or methyl isobutyl ketone) contaminates the burner.

Solutions: Clean the burner

4) When instrument fails, try to use copper, magnesium or other elements lamps with an absorption wavelength at 280nm-350nm and little influence of inner flame condition on absorption to see if there are any improvements.

5) Lamp current, slit and flow of acetylene and combustion-supporting gas are improperly set.

6) Waste solution pipe is not in proper condition with improper solution discharging.

- 7) End of atomizer is polluted.
- 8) Atomizer is adjusted improperly with too big fog drops. 9)

Output pressure of acetylene cylinder is less than 0.06MPa. 10)

Output pressure of air compressor is less than 0.3 MPa.

11) Check air filter, specially when over amount of sodium emission is found in the flame.

Impure acetylene will produce such kind of emission, too.

10.7 Dynamic Drift

The instrument adopts a double beam optical path. The signal processing circuit can automatically compensate the zero point drift caused by the drift of element lamp emission intensity.

Possible causes of dynamic drift (referring to zero point drift in the practical measurement state):

1) Burner has not been pre-warmed (Formal measurement shall be started after ejection of blank solution for 5 minute to pre-warm the instrument).

- 2) Sample-in capillary tube may be blocked.
- 3) Hollow cathode lamp is improperly placed on the holder.
- 4) Too big scale expansion leads to the expansion of zero point drift.

10.8 Reading Drift or Poor Repeatability

Causes :

1) The burner has not been pre-warmed for enough time. 2)

Burner slit or atomizer capillary tube is blocked.

3) Atomizer capillary tube has leaks. 4)

Atomizer capillary tube is polluted.

5) Waste solution outlet is blocked or soaked in the waster liquid. It leads to water accumulated in the combustion chamber.

- 6) Gas working pressure changes.
- 7) Temperature of the sample to be measured changes.

10.9 Firing Difficulty

Causes :

1) Pressure or flow of acetylene is too small. 2)

Flow of auxiliary gas is too big.

3) When the instrument has been stopped for a long time (overnight or longer time), air

disperses to fill up the flame lighter's channel. A relative long firing period shall be used (several times of firing) to make acetylene to fill up again the channel.

10.10 Flashback of Burner

Causes :

- 1) Direct fire_acetylene flame
- 2) Water sealing of waste solution discharge pipe is improperly installed.

Attached Table1 1: Atomic Absorption Standard Conditions

Eleme	WL (nm)	BW (nm)	FG	SC (a)	Elemen	WL (nm)	BW (nm)	FG	SC (a)
Ag	328.1	0.7	A - C	3	Ni	232.0	0.2	A - C	7
As	193.7	0.7	A - C	40	Pb	283.3	0.7	A - C	25
As	193.7	0.7	Ar - H	7	Pb	247.6	0.2	A - C	12
Au	242.8	0.7	A - C	12	Pt	265.9	0.7	A - C	90
Ba Bi	553.6 223.1 422.7	0.2 0.2 0.7	A - C A - C A - C	225(b) 18 3.5	Rb Rh	780.0 343.5	4.0 0.2 0.2	A - C A - C	5(b) 15 25
Ca	422.7	0.7	A - C	3.5	Ru	349.9	0.2	A - C	25
Cd	228.7	0.7	A - C	1.2	Sb	217.6	0.2	A - C	25
Co	240.7	0.2	A - C	7	Se	196.0	2.0	A - C	25
Cr	357.9	0.7	A - C	4	Se	196.0	2.0	Ar — H	12
Cs	852.1	4.0	A - C	15(b)	Sn	286.3	0.7	A — C	180
Cu	324.7	0.7	A - C	4	Sn	286.3	0.7	A — H	70
Fe	248.3	0.2	A – C	6	Sr	460.7	0.14	A – C	6(b)
Hg	253.6	0.7	A – C	350	Tc	261.5	0.2	A – C	120
ln	303.9	0.7	A - C	35	Te	214.3	0.2	A - C	45
Ir	264.0	0.2	A - C	400	Ti	276.8	0.7	A - C	25
K	766.5	4.0	A - C	1.8	Zn	213.9	0.7	A - C	0.8
Li Mg Mn	670.8 285.2 279.5	1.4 0.7 0.2	A - C A - C A - C	1.5 0.3 2.5					
Mo Na	313.3 589.0	0.7 0.14	A – C A – C	40 0.7					

(a) The value refers to water solution sample concentration $(\mu g/ml)$ at the absorptance of 0.2. (b) Add large amount of easily ionizing substance (KCl 100mg/ml) to control ionization.

c) The sensitivity is reached after addition offluoride.

d) WL- Wavelength, BW-Bandwidth, FG- Flame Gas, SC-Sensitivity Check

e) A- Air, C- Acetylene, Ar-Argon, H-Hydrogen

Element	Sensitivity Check * +	Element	Sensitivity Check * +
Al	0.05	Pb	0.025(283.3nm)
As	0.04	Pb	0.016(217.0nm)
Au	0.04	Pd	0.04
Ва	0.03	Pt	0.18
Bi	0.02	Sb	0.04
Cd	0.003	Se	0.06
Со	0.03	Si	0.14
Cr	0.007	Sn	0.14 (286.3nm)
Cu	0.01	Sn	0.08(224.6nm)
Fe	0.01	Те	0.025
In	0.05	Ti	0.21
lr	0.3	V	0.15
Mn	0.005	Zn	0.0007
Мо	0.025		
Ni	0.04		

Attached Table 2 Graphite Furnace Analytical Sensitivity Check

* All data are obtained using graphite tube coated with thermal medium under the condition of fast increase of temperature and stop of gas.

+ The value refers to the metal concentration $(\mu g/ml)$ of water solution at the absorptance of 0.2 with the sample-in amount of 20 μ l. The more the sample-in amount is, the higher the sensitivity is.

Note: Data in the attached table 1 and 2 are adopted from PERKIN-ELMER Instrument Materials.

11. Running Environment

AAAXP is a PC-CRT data processing software specifically designed for AA320 and AA320N series.

The following are AAAXP's requirements to a computer system:

Hardware requirements:	CPU:	>Pentium IV or above
	RAM:	>256 MB or above
	Special A/D card	
	Special I/O card (graphite oven is optional)	
Software requirements:	Windows XP (English Version) or above	
	Microsof	t Office Access

12. Software Installation

1. AAAXP contains one installation disk.Run SETUP, change installation disks or type necessary information prompted by instructions on the screen(as in 1) to complete the installation.

After the installation is accomplished, the following 4 files will be seen in the installation path(folder AAAXP):

AAAXP.DEP AAAXP AnaCon St5unst.log

13. What We Will Do

Suppose users analyze on a simple atom absorption spectrometer. Having prepared the standard solution and the sample solution, the operator usually needs to accomplish the measuring report after a series of procedures. The procedures are following:

- Look up the element that needs to be analyzed in the periodic table of elements, in order to get the necessary analytical conditions such as its wavelength, slot, flame, or the rising temperature procedure of the graphite oven to set up the spectrometer's host machine and the graphite oven.
- Choose the measuring conditions such as the method of measurement (integral, peak height, peak area), time, number(average), background correction, etc, in order to get reliable analytical result.
- 3. Measure the standard solution; draw, test and modify the working curve.
- Measure the solution that needs to be measured, find the corresponding concentration value, modify the measurement, and record the result.
- Calculate the percentage composition, display the data and summarize the data as a report form and print it out or save it in the archives.
- 6. Record and file the satisfying results obtained from the analytical conditions and measuring conditions in this operation for the next examination.

AAAXP is meant to apply computerized technique to aid operators to finish the above routine jobs, and all these aids are to be done by a series of function windows. These windows will be opened one by one according to the job procedure upon the operator's order; therefore, it's easy to use them. In order to help the readers understand the contents while reading, please launch AAAXP in the meantime so as that the screen will be the illustration to the content. Now let's start.

14. Starting Operation

14.1 To start AAAXP

Turn on the computer, and double click AAAXP icon on Windows95/98 desktop. Users see the startup screen of AAAXP in the middle of the desktop, which means AAAXP starts successfully. A few moment later users automatically enter AAAXP system window.

14.2 System Window

System window is the container and administrator of all the function window. All the programs in the system are operated in this window.

Starting from the top of the screen, the first line is title bar; second menu bar, and then tool bar. These bars and the space under them become AAAXP system (users start AAAXP and initially enter the system window, and that space is occupied by the element opened automatically and mode).The three bars of system window always display during the whole operation, but the window could be blank or occupied by one or more function windows. Please note Windows taskbar is kept on the bottom of the screen.

On the left of <u>Title bar</u> shows the name of this software and its icon, on the right of which are three operating buttons, referring to "Minimize", "Restore", "Close". Single click "Minimize", and the system window can be minimized as an icon in the Windows taskbar(Single click it again to restore the system window). Single click "Close" to shut down the system and quit the operation.

There are three pull-down menus "File", "Window" and "Help" on the menu bar (the letters are shortcuts). From "File" users may choose the current function window of the system: element and mode, condition settings, sample measuring, file management or the maintenance of analytical conditions library. They can be opened separately or at the same time. From "Window" users may choose the arranging pattern of the opened function window; and read this instruction in "Help".

The four command buttons with icons on the left of <u>Toolbar</u> are used to choose four major function windows: element and mode, condition settings, sample measuring and file management. The 2 columns in the middle show respectively the current work mode and analytical element. When users start AAAXP, the system automatically sets the mode as the flame atom absorption, and the analytical element as Cu.

Click "Close" on the top right corner on the System anytime to exit AAAXP.

14.3 Element and Mode Election Window

Open: 1. Starting AAAXP and entering System automatically opens this window.

2. Single click "File" on the menu bar on the system window and choose this window from the pull-down menu, or click the 1st icon on the tool bar, to open this window.

Close: Single click "Close" on the top right corner of this window.

Function: Choose the analytical element and mode.

Please note the icons and names on this window's title bar. The three operating buttons' functions are similar as the ones in the system window.

The atoms are arranged in the middle of the window according to the periodic table of elements, in which 68 elements may be applied in flame atomic absorption analysis. The types of fire which can be applied on them are colored, for those which cannot be analyzed are marked with grey. 11 elements such as As, Ba, Ca, Cr can be applied with 2 types of fire analysis, and this software provides the fire with lower sensitivity interference (users may change it in the Analysis Conditions Maintenance). The color marks have nothing to do with the fires out of the atom emission.

There's a box displaying pre-elective elements on the top of the periodic table of elements. There are a set of mode selection buttons and <Confirm>and <Cancel> on the bottom.

How to choose Elements and Mode? The first step is to click the element's name to display it in

the element pre-selective box; then click the necessary mode, finally press <Confirm> (the warning sign after pressing is to avoid losing measuring data for not saving it). The new mode and element are shown in the system window after these procedures, while their analysis conditions are read from the condition library and put into condition setting window.

NOTE 1. If users only change the element without doing the mode, the analytical condition of the previous element will not be changed; otherwise all the state of the system will be changed.

2. Setting up will be effective after clicking <Confirm>.

14.4 Condition Setting Window

Open: Click "File" on the menu bar of the system and choose Condition Setting window, or click the 2nd icon on the tool bar, to open this window.

Close: Single click <Close> on the top right corner of this window.

Function: Display the analytical conditions of current element as well as mode of the spectrograph's host machine (graphite oven)as a reference to the settings.

Note the icons and names on the tool bar of this window, and the three buttons of the window have similar function as the ones in system window.

Note the two choice pages on their pages. Single-click any TAG labels can bring this page to front.

Analytical Condition Window

The system will automatically find analytical conditions from Analytical Conditions Library according to difference elements, and show them on the page, as a reference to set the spectrograph's host machine(graphite oven).Users may change the contents of all the columns according to the actual condition so as to save the files, and the changes do not affect the data in the Analytical Condition Library. Read 15.8 Maintenance of Analytical Condition Library if users want to change the analytical conditions permanently. "High-voltage", "Sample light" and "Reference Beam" refer respectively to the actual high voltage value and energy level on the spectrograph. When the host machine of the spectrograph captures the desired wavelength and find the optical alignment, "High Voltage" should be adjusted to maintain the energy level of "Sample light" and "Reference Beam" at around 80(there is sure to be differences between the two. Less differences mean higher stability of the reading).

Measuring Conditions

Signals and data procedure can be set on the page. Choose the measuring times for average values (repetition), reading significant digit (precision), and measuring period (measuring). Press the button and wait for a few seconds for the stable reading (time lag), so as to get satisfying analytical results.

The measuring results (total absorption-background) and background are to be listed at the same time after "Background Adjustment" is set.

When "Repetition" is more than one, the measuring result lists the average value, standard deviation and relative standard deviation (coefficient of variation).

How to set up concentration ? The following should be done to communicate with the computer after "Concentration" is chosen:

1. Choose the types of specification curves:

Rectilinear regression: The least squares technique mathematical model is able to ensure the standard average inaccuracy, but can not be sure to make the curve pass zero and reference mark. When user makes sure the samples' concentration can be chosen within the linear range. The slope, intercept and linear related coefficient are set at the same time when working curves are set. Slope should be re-adjusted for sensitivity drift resulting from long-time measuring.

Curve fitting: Special conic mathematical model. The curve passes zero and each of the reference mark and user may choose it when the sample's higher concentration may make concentration/absorption curves curve. Re-adjusting slopes may cause more inaccuracy.

Standard mixing: suitable for marking and measuring the unknown sample with complex substrate. Mix the unknown sample into the standard solution with known concentration into the unknown sample to make a series of solution of nominal concentration. Unknown sample is sample no.1, with nominal concentration 0. Rectilinear regression working curves can be obtained after special mathematical treatment.

NOTE If users cannot decide when choosing rectilinear regression or curve fitting, they may choose rectilinear regression, because there's still one chance to choose after finishing rating curve(See <u>15.5</u> <u>Sample Measuring/Rating curve</u>).

2. Choose Units of Concentration.

3. Input Standard Concentration:

Parameters can be put into "Standard Concentration" only when "Concentration" measuring method is set. Input a series of instantizing titrating solution concentration one by one in the text frame above and press <Enter> to confirm in the meantime. Standard concentration is to be listed in the ascending order on the middle list frame, and standard numbers are recorded in the list frame on the bottom. Double click the chosen standard concentration to delete the value. 10 standard concentrations can be listed in the system at the same time, which means the maximum reference mark on the working curve is 10.

NOTE The procedure to input standard concentration is slightly different: The concentration of the unknown sample, as standard 1, is set to 0, and then a series of standard concentration based on the same unknown sample is input accordingly. After the actual marking is set, the system will automatically get each of the standard actual concentration including the unknown sample and set up the new curve. Then samples of the similar substrate can be tested.

NOTE: 1. When the standard curve is linear regression or curve fitting, the standard concentration can not be 0. The first standard concentration has to be 0 for the standard inputting methods.

2. The working curve has not been set up yet in this way. The standard solution must be tested in Sample Measuring, and all the values of absorbency should be obtained according to all the standards, then the system is able to set up a real working curve according to the chosen curve.

3. Click <Confirm> on the bottom-left corner of the window to make all the settings take effect.

14.5 Sample Testing Window

Open: Click "File" on the menu bar of the system menu and choose this window's name on the pulldown menu, or click the 3rd icon on the tool bar to open this window. **Close:** Single-click <Close> on the top right corner of this window.

Function: Measure and display the data.

Users should have chosen the condition in the previous window before entering this one, and set up the photometer host machine and prepared the solution for measuring. As the routine ordinary operation, users now need to input the samples according to the serial numbers and press the buttons. The major work of measuring samples is to be done on the Sample Measurement of the window.

Usually blank solution is inhaled and the reading is set to 0 as the datum mark before the official sample is measured. If measuring "Concentration" is chosen, the samples should be input according to the standard serial number order to finish marking the working curves. If the step is repeated for several times (for the average), each sample should be measured several times successively.

NOTE the icons and names on the tool bar of this window, and the three buttons of the window have similar functions as the system window.

Note the three choice pages on their pages, paying attention to their names on the tag. Single-click

any TAG labels can bring this page to front.

Sample Measuring

The left side of this page is the data frame which rolls to record measuring results, and the right side is the major display column for showing the simultaneous measuring results. 7 function buttons and 1 state frame are listed under displaying frame. The functions are following:

<Zero Setting> Click and start measuring to automatically set the result to zero, and setting and the measuring result is not kept. Starting under the condition of not inhale sample to set the spectrograph's mechanical zero as checking basic linear stability. Starting under the condition of inhaling the blank solution may set measuring zero.

<Measuring> Click and set up a measuring, the measuring result is saved in memory, and the list is displayed in the data column on the left.

NOTE:

The reading circles of zero setting and measuring are decided by the <u>Condition Setting Window</u>.
 During measuring, the main column counts the time.

2. Inhaling the sample, solution and press the button until the major reading is stable when zero and measuring are set. The capillary can not be put out before the result is displayed, otherwise there will cause some errors.

3. If the mode is absorption by the atoms in the graphite oven, two buttons <Cancel Waiting> and <Cancel Operation> will be displayed on the top right of the window after starting the two buttons, because when the graphite oven is operated, the system will send a signal to start heating up program to the oven and wait the graphite oven to send a signal back to start system readings. "Cancel Waiting" means waiting no more for the signals from the graphite oven and start the system; while "Cancel Operation" means stopping the present zero setting or measuring.

4. Click the zero setting or measuring button, a dotted line frame will be displayed out of the button's name, meaning this button has become the default command button of this window. Pressing <Enter> or space on the keyboard may start zero setting or measuring, so that users may operate on the keyboard to measure. Other buttons on this window will obtain the function of default command.

<Delete> used to delete the measuring results for all the unknown samples. Caution! <Modify> used to re-measure the measured results and replace the old results with the new ones. Click the button and a "?" will be displayed at the cursor. Move it to the data to be modified. Double click and the data will disappear immediately and be refilled by the newly measured results.

<Curve> used to enter Rating Curve page (See Rating Curve)to check working curves. The button turns grey before the working curve is marked, meaning that this button cannot be used under the present condition. The button turns normal after the working curve has marked.

<Slope> the complete form is Slope Reset, used to correct sensitivity drift when measuring concentration. Spectrograph's sensitivity may change for some reason after working for a period of time(called Sensitivity Drift), which causes troubles for the concentration of unknown sample obtained with the former working curve. There are 2 solutions: one is to completely reconstruct the working curve, but a series of standards need to be re-measured, which means a lot of work. The other(useful for linear working curves) is to measure only one standard, and match the result with the former result obtained from the same standard, to get offset coefficient, and use it to modify the working curve and the every result afterward. Mathematically it means to reset the slope of the linear equation.

This system regulates the slope needs to be re-measured under the high-level concentration. The processes are: inhaling the standard solution of the highest concentration marked for the previous time, then click this button to start measuring. This button is grey before the first working curve is marked and cannot be used.

<Print> Press this button to turn on the printer, and print out the measuring result.

<Input> a state button. Press it to change auto sampling to the photometer by the computer to type the new data to the computer. Press and then the major display frame turns blue and a cursor appears. Then type absorbency(or emission power), press <Enter> to confirm, to simulate computer's auto sampling. Press this button again and restore.

Signal Waveform

Persistent Waveform: if the desktop grapher recording meter shows real time continuous measuring signals. Choosing appropriate measuring scale, the spectral line of emission of element lamp can be scanned or the baseline stability of the spectrograph can be shown and printed. The graph of persistent waveform is draw by the computer with auto sampling.

Reading Waveform: Capture the signal's dynamic waveform during the measuring circle. It's especially suitable for observe the peak form waveform signal of the graphite oven atomization to choose suitable ladder temperature. The time measuring scale is decided by the measuring time in the Condition Settings. The graph of reading waveform is drawn after pressing <Measure> by the computer after sampling.

Both the two pages above have a graph frame to display signal waveforms, among which the longitudinal coordinate is absorbency or emission power(according to the mode), horizontal ordinate is time. The time unit of persistent waveform is minute (min.), and the time unit of reading waveform is second (sec.). If background adjustment is chosen in the Condition Settings, the background curve turns out to be red in the Waveform window in the meantime.

On the bottom of the graph frame are button groups, among which <Zero Setting> and <Measure> have the same functions as the button of the sample measuring page. The measuring results are recorded in memory. The functions of other buttons are the following:

<Measuring Scale> changes the measuring scale of graph display to satisfy the needs for measuring. The time unit of persistent waveform is minute, and the time unit of reading waveform is second. Press the button and a dialogue box appears in the middle of the page for inputting.

The time measuring scale of reading waveform is decided by the measuring time in the Condition Settings and cannot be changed on this page.

<Refresh> clears the present graph display, re-sample and draw the picture.

<Print> Print the present graph.

Rating Curve

This page shows the data and graph of rating curve. Click <Curve> on the <u>sample measuring</u> page to enter this page. The right side of the page is the measuring data of the standard solution while the left side is the graph of working curve with the type of the graph. If it is a linear working curve, the slope, intercept and linear relative coefficient are indicated.

The functions of the buttons are the following:

<Variation Curve> Draw the working curves of the other type with the same group of measuring data and offer them to the users to choose, if the linear regression is changed to curve fitting or the contrary. This function allows the users not to have to worry too much to choose the type of specification curve on the <u>Condition Settings</u> page, because there is still one more chance to choose after the curve is drawn.

NOTE: Though the curve can be changed for many times, the working curve displayed for the last time (before exiting the program) is the working curve truly operated when the unknown sample is measured.

<Print Curve> Print the data and graph displayed on the present page.

<Exit> Return from this page to <u>Sample Measuring</u> page.

14.6 File Management Window

Open: Click "File" on the menu bar of the System and choose this window's name on the pull-down menu, or click the 4th icon on the tool bar to open this window.

Close: Single click "Close" on the top right corner of this window.

Function: 1. Save the data as a new file or to an existing file when finishing the data maintenance after measuring.

2. File management.

Please note the icons and names on the title bar of this window. The functions of the three operating buttons are similar as the ones in the system window. Open this window to enter File Management page. The existing files are listed in the data file catalog frame, the left side is the reference column of chosen files. On the bottom of the data file catalog frame is a group of operating buttons along with present path display columns.

The file management strategy of this system is: a file management library named "FileManage.mdb" is used to save all the management data files of the appointed path. Every data file may contain several different elements' measuring data, in order to save the data of the different analysis element of the same samples in the same data file.

If users enter this page, the system will automatically look for "FileManage.mdb" in the system path. If there is a path then it'll be opened automatically, and a path will be created automatically if not. The data files in the file management library will be listed in the "Data File Catalog" frame, and the blue on filename means the file is selected(you may select it with a mouse). "Reference" column shows the reference of the selected files. Further operation can be realized with the following

buttons:

<Save File> Save all the measuring data of the present system into the appointed file and put it into the current file management library. Press the button and a save file frame appears. Users can choose to save into a new file or an old one (the former names a new file while the latter saves the data of the different analytical elements of the same samples in the same data file. If the saved is the first of database then there is no need to choose), then input in the accordingly column the file name (a must), reference (optional)and operator mark(optional), and finally press <Save>.

Note: Chinese is allowed to use in this system if single-click the En icon in the Windows task bar on the system window and choose the input method.

<Load File> focuses on the chosen file and load it to update the present system. Single-click the button and a dialogue of choosing loading elements appear on the page, showing **file name** and the elements it contains. Choose one of them and single click <Confirm>, then the system is updated by all data of this element. The button is grey and invalid when there's no file in the data base.

<Delete File> focuses the selected files and delete the selected files. This button is grey and invalid when there is no file in the data base.

<View File> focuses on the chosen file. Press the button, open View Data (see <u>15.7 View Data</u>), and browse the data saved in the appointed file. This button is grey and invalid when there is no file in the data base.

Modify the path appointed by "Current Path" to create or open the file management library under any path.

14.7 Data browse Window

Open: Click <Browse File> in file management page to open this window.

Close: Single-click "Close" on the top right corner of this window.

Function: Browse the data saved in the appointed data file on the file management page.

Please note the icons and names on the title bar of this window. The functions of the three operating button are similar as the ones of the system window.

Note the 4 choice pages in the window, paying attention to their names on the tag. Single-click any TAG labels can bring this page to front. The functions of each page are the following:

Analysis and Measuring Conditions:

This window shows the analytical and measuring conditions of the current elements in the files. On the bottom of the window list from left to right 4 icons with arrows, showing <to the first element>, <to the previous element>, <to the next element>, <to the last element>.

Measuring Data:

This function shows the measuring data of the present element in the file. Under the display frame there is another button <Display All Data>. Press this button and all the measuring results of the current element, including repeated data, will be displayed; otherwise only the effective measuring results will be displayed. The buttons on the bottom of the window are the same as analytical and measuring condition page.

Sample Summary:

This function summarizes and displays all the elements' concentration and percentage concentration(percentage concentration) in the file. Click <Percentage Concentration> on the button of the page and shift to percentage concentration calculating page to calculate.

Percentage Concentration Calculation

The left top of the page is the options of users samples, and the right side lists the calculating formula of percentage concentration(for testing calculating results). The buttons are 3 data input

column.

Users should choose the sample preparation method and input according elections the data of "Sample Quality", "Volume of Mother Solution", "Dilution Rate". For example, sample preparation method is "the quality of the samples is different while the sample solution of the same element has the dilution rate". In order to input a group of 3 samples and each sample's data from testing copper and iron, 1 volume will be input into "Volume of Mother Solution" frame, and copper and iron's dilution rate in the "Dilution Rate". Press <Enter> or space to confirm after inputting any value. System will count automatically and prompt the next input object. When some column is finished according to the requirements, the column turns blue. Press <Calculate> after inputting all the data. The calculating result will be shown on the sample summary page.

Generate Report Forms:

Users can choose to generate report forms on this page and print analysis report forms file. <Condition Record> and <Measuring Data> focus on the data on the files of analytical/measuring condition page and measuring data pages(the data on the measuring of the single element), while <Sample Concentration> and <Percentage Concentration> will print all the elements' data summary in the files. Therefore, these two options and the other two are not compatible. For every report forms printed, a title can be made in the "report forms title" column. Chinese is allowed to use in the title, if only single click the En icon on the Window task bar and choose the input method.

14.8 Maintenance of Analytical Condition Library

Open: Click <File> on the menu bar of the system window and select to open this window.

Close: Single click <Close> on the top right corner of this window.

Function: Maintenance of the analytical condition library.

Analytical condition library includes three databases: "flame atom absorption", "graphite oven atom absorption" and "flame atomic emission", all of which show the settings of the host machine of the

spectrograph and the controller of graphite oven in the Condition Settings according to the mode and analytical elements chosen. Since the actual analytic samples are various, the analytical requirements are different; therefore, the conditions from the database are unavoidably limited. Users may change the conditions according to the actual settings on the condition setting windows, and change the conditions through this window permanently.

Please note the icons and names in the title bar of this window. The functions of the 3 window operating buttons are similar to the system window.

After users open this window, the system automatically opens "flame atom absorption analytical condition library". The sum record marked records the current groups of data in the library (the analytical condition of one element is one group of data), "current record" indicates the number of the current data (the number has nothing to do with the elements' number). In the middle of the page is a group of analytical conditions for the current elements. The 4 buttons with arrows on the bottom of the window show from left to right: <to the first element>,<to the previous element>,<to the next element>,<to the last element>. And the functions of other 4 buttons are the following:

<Modify> Pressing it means users want to modify the data for the current record, and they may do it by clicking into any condition column.

<Add> Pressing it means users want to add one or more elements' analytical conditions, and the system will automatically shift to the latest record number, open a group of blank condition columns for the users to add new records.

<Delete> Pressing it means users want to delete the data of the current record. If confirm to the cautious prompt, current record will be deleted completely. Caution!

<Renew> Press it after modifying or adding records. The system will save the modified or added records into the database.

On the right side of the page list 3 buttons of "condition library".

The arrangement and operating method of the pages "Graphite oven atom absorption" and "flame atomic emission" are the same as the "flame atom absorption" above.

NOTE: Users may input Chinese messages into the condition library, only if they single click En icon on the Windows task bar of the system window and choose input method.

15. Trouble Shooting

Trouble: The system does not respond when choosing or changing working condition or parameter.
 Solution: Users do not press <Confirm> after choosing or changing working condition or parameter;
 therefore, the system still keep the same.

2. Trouble: The values of high voltage, sample and reference in the analytical condition window are all shown as full, while there is not any absorption response.

Solution: A/D card error. Commonly it's because the address switch is set wrong or one of the switches fails to work. The correct settings are: OFF, ON, OFF, ON, ON, ON, ON, ON. (OFF means disconnection, and ON means connection.) Users may test with multimeter to see if the switch block fails to work and fix it.

3. Trouble: The values of high voltage, sample and reference in the analytical condition window are all shown as full, while there is not any absorption response.

Solution: Signal transportation error. Check if the signal wares are connected correctly.

Trouble: Data have been input into the columns of percentage concentration window, but
 <calculate> fails to be pressed.

Solution: Users fail to press <Enter>to confirm after inputting the data or data inputting is not finished yet. Three columns will be blue if all the data is input.

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