ASC-990
Graphite Furnace
Autosampler

INSTRUCTION
MANUAL

Contents

❖

Chapter 1 Introduction

1.1.   Introduction

The device is a dedicated autosampler for Atomic Absorption Spectrophotometer. It can sip standards and samples automatically and clean the sampling head and pipelines automatically as well as add matrix modifier etc solvents automatically. Its look is shown as Figure 1-1.
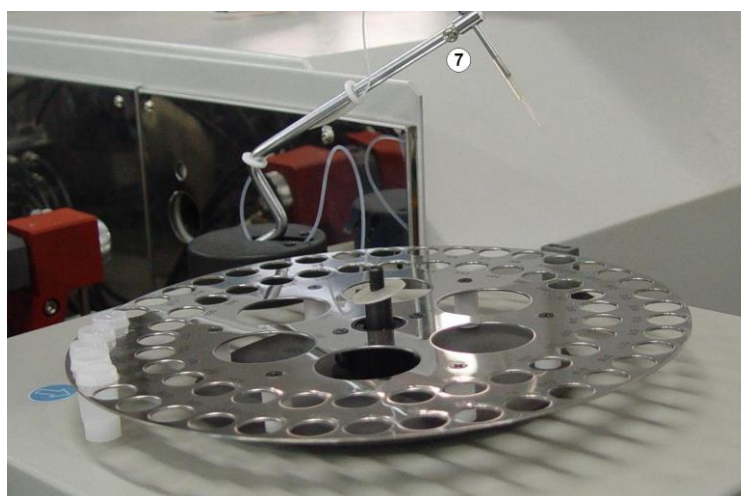

Figure 1-1: Profile

The construction of autosampler is described as bellow:
1. AC POWER INLET
2. Power Switch: Power ON/OFF
3. RS-232-C CONNECTOR: Connected to PC COMM PORT
4. 485 INTERFACE: Reserved
5.WASH SOLUTION CONTAINER
6. SHOE PLATE of autosampler

❖

Side View

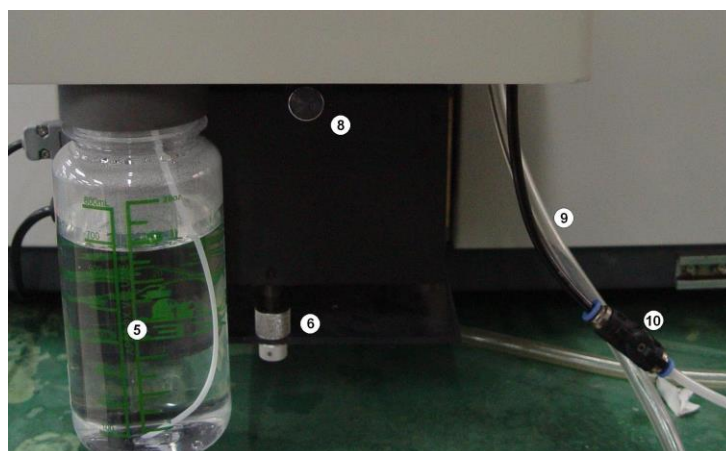7. SET SCREW on sampling arm: To adjust the length of sampling arm.

Top View

8. ALLOCATION SCREW: To adjust the back and forth position of autosampler.
9. DRAIN HOSE of autosampler: Another end of it should be put into a waste liquid container.
10. ARGON INTAKE TUBE and BALL VAVLE: The autosampler uses Argon Gas to drive the wash solution from its container. A ball valve is used for control of Argon Gas supply. Another end of it should be connected with the ARGON INTAKE TUBE of the furnace power supply unit through a TRIPLE VALVE.

Front view

1.2.　　Environment requirements

1．Power supply: AC 220V±10% 50HZ 50W
2．Ambient temperature: 5-40℃
3．Ambient humidity: less than 70%
4．Communication port: RS232
5．Software:　AAWIN2.0 or later

Note: The communication ports of the computer, main unit and the autosamper are RS232 interfaces. So the computer should have two RS232 interfaces, or one RS232 and one USB interface. A USB-to-RS232 adapter is needed when USB-interface is used. The 485 interface on the autosampler is designed for factory-use. A user couldn't use it.

Chapter 2 Installation

2.1 Preparation

● After the device has been unpacked, please check up carefully the parts provided against the packing list for acceptance.
● Read the instruction manual carefully to be familiar with the installation procedures and operation.

2.2 Installation and adjustment

1．On UVWIN software, select "Measure Method" to be "Graphite Furnace" (if "Graphite Furnace" was set, skip the step).
2．Put on a graphite tube, and adjust the position of the graphite furnace, making it at the best position in the optical path.
3．Click on the icon " Autosampler" in the tool bar to enter into the window of autosampler setup. See Fig. 2-1.
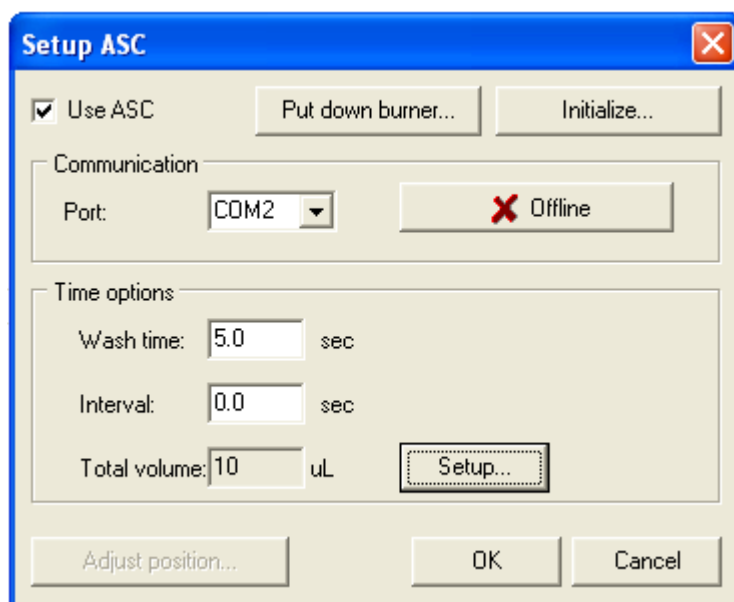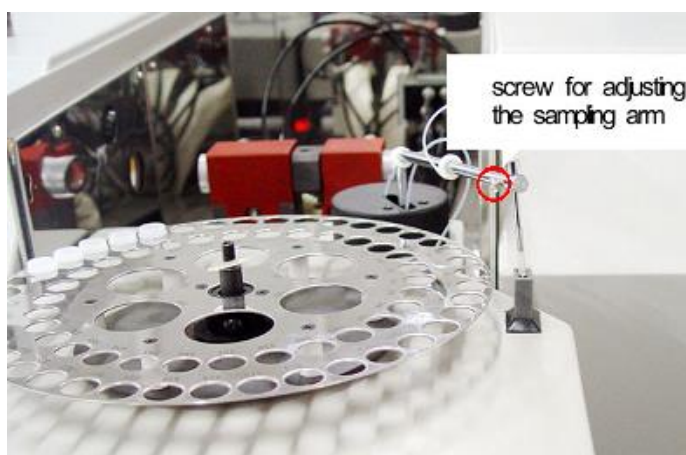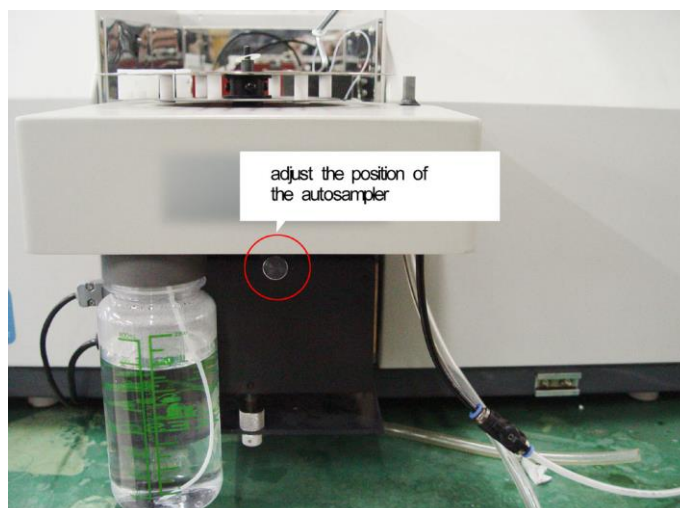

Fig. 2-1 Setting autosampler

4．Click on the button of "Put down burner" to move the flame burner to low limit (if an autosampler is mounted, skip over 4-9).
5．Remove the burner, the tray under the door of combustion chamber, and the capillary of the nebulizer. Then rotate the nebulizer counterclockwise around 90°.

❖

6．Install and fix the shoe plate of autosampler.

7．Install and fix the autosampler. Be sure the power of autosampler is off, and then rotate the sampling arm till it is above the clean slot. Adjust the length of the sampling arm to make the capillary to be able to insert into the hole of the cleaning slot, and then tighten the screw on the sampling arm.

8．Rotate the sampling arm above the graphite tube, then adjust the back and forth position of autosampler to make the capillary to be able to insert into the graphite tube. (Lift up and put down the sampling arm repeatedly with a hand, and observe whether the capillary can insert into the graphite tube.)

9．Connect a power cord and a communication cable for autosampler, and ensure the autosampler connects to the RS232 interface of PC .

10. Please refer to Fig.2-2 to observe the capillary. Make sure it is treated properly as required in the figure.
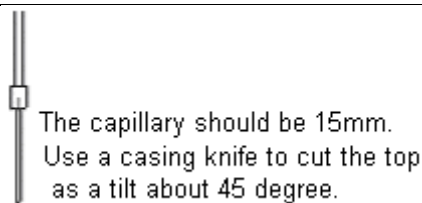
The capillary should be 15mm.
Use a casing knife to cut the top
as a tilt about 45 degree.

Fig. 2-2 Requirements of the capillary

11. Switch on the power of autosampler, and check the check box of "Use ASC", to make it showing " √ ".
12. Select the communication port corresponding to that of autosampler from the pull-down list of communication port.
13. Click on the button of "Offline" to start connecting. After it connects with PC, it will display "online".
14. Input "wash time". "5-10" is recommended. Viscous samples need longer wash time.。
15. Input "interval" for repeating sampling. "0" is recommended.
16. Click on the button of "Setup" designed for "total volume" to enter into the window of setting mix sampling. See Fig. 2-3. The window is used for setting sampling volumes of samples and additional matrix modifier.
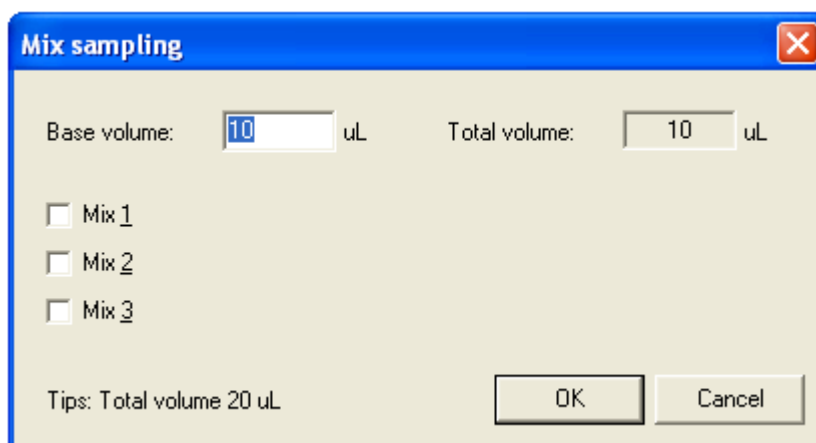

Fig. 2-3: setting the mix sampling

17. Input for "Base volume", that is, sampling volume of samples. Its range is 5-20ul。
18. Sample mixing with no more than 3 kinds of solution is available. First, select a "Mix" sample, to make its check box marked " √ ". Then, select the sample tray position from the pull-down list of "position". Then input its "volume". After finishing all settings of the sample volumes, it will automatically calculate the "total volume". Click on the button of "OK" to return to the former window of setting autosampler (Fig.2-1).
19. After the button of "Adjust position" is clicked, the sampling arm will

❖

automatically move to the position above the graphite tube, that is, sampling position.

20. Lift up the sampling arm with a hand. Click the button of "Lift up" to keep it not going down. Then observe the position of capillary relative to the graphite tube. Click on the buttons of "Right" or "Left" till the capillary can enter into the graphite tube easily. To do the adjustment, you 'd better to get other person's assist, to adjust the back and forth position of autosampler accordingly.

Fig. 2-1 Position adjustment

21. Put down the sampling arm to let the capillary enter into the graphite tube. Then use a mirror to observe from the right side of furnace the insertion of capillary into a graphite tube. Click on the buttons of "up" or "down" to make the capillary just reach to the bottom of graphite tube. Click on the buttons of "Lift up" and "Put down" repeatedly till you get a satisfactory position. Then close the window.

22. Connect the Argon Intake Tube of autosampler with a Ball Valve and a Triple Valve to the Argon Intake Tube of Furnace Power Unit. From the Triple Valve, one tube goes to the Argon Cylinder, one to the Furnace Power Unit, and one to the Furnace Autosampler.

23. Connect the DRAIN HOSE of autosampler and put its end into a waste liquid container.

Note: You should repeat the steps of 19-21 if any changes happen to the mechanical position of autosampler or to the capillary.

❖

Chapter 3 Operation

3.1 Setting sample wizard

Before measuring, you need to make settings for samples being measures. Select "configuration" → "sample wizard" or click the button " $\overset{\text{\tiny\textbf{※}}}{}$ ", you can get the sample wizard window.

"Correction method": Select a correction method for standards. Three methods are available: "calibration curve", "standard addition", and "interpolation".

"Curve equation" is a calculation equation of standards. The equations available are: "1st Order: $[A]=K_1[C]+K_0$", "1st Order: $[C]=K_1[A]+K_0$", "2nd Order: $[C]=K_2[A]^2+K_1[A]+K_0$", and "3rd Order: $[C]=K_3[A]^3+K_2[A]^2+K_1[A]+K_0$".

"Conc. Unit" is the concentration unit of samples. The available units are: "ng/ul", "ng/ml", "ug/ul", "ug/ml", "mg/l", "ppb", "ppm", and "mol/l". If the concentration unit you need is not included, you can input it.

"Sample Name" and "Start ID" compose a sample ID. For example, if a sample ID is "STD1", its "Sample Name" is "STD" and its "Start ID" is "1".

Fig. 3-1 Setting the sample wizard

After finishing the settings of standards parameters, you can click "next" to enter into the window of setting standards concentration. See Fig. 3-2.

❖

1．Input each concentration for standards in the column of "Conc." Of the standards list. Click "Add" to add a standard, up to the most of eight. Click "Delete" to delete a standard, down to the least of one.

2．In the pull-down list of "Initiative position", select the start position of standards placed on the tray of Autosampler. The default is "ST1".
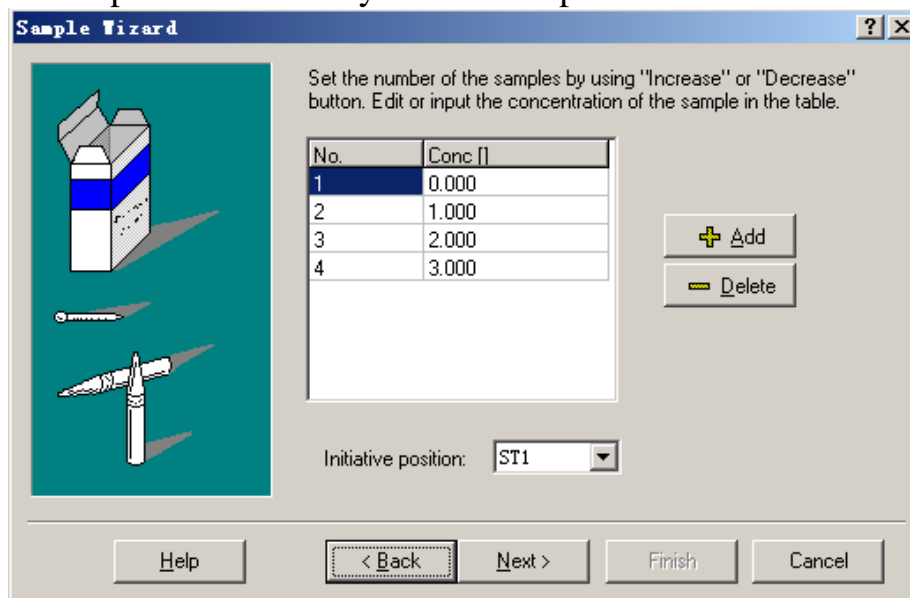

Fig. 3-2 Setting concentrations

After finishing setting of standards concentrations you can click "Next" to enter into the window of auto-analysis mode, see Fig.3-3.
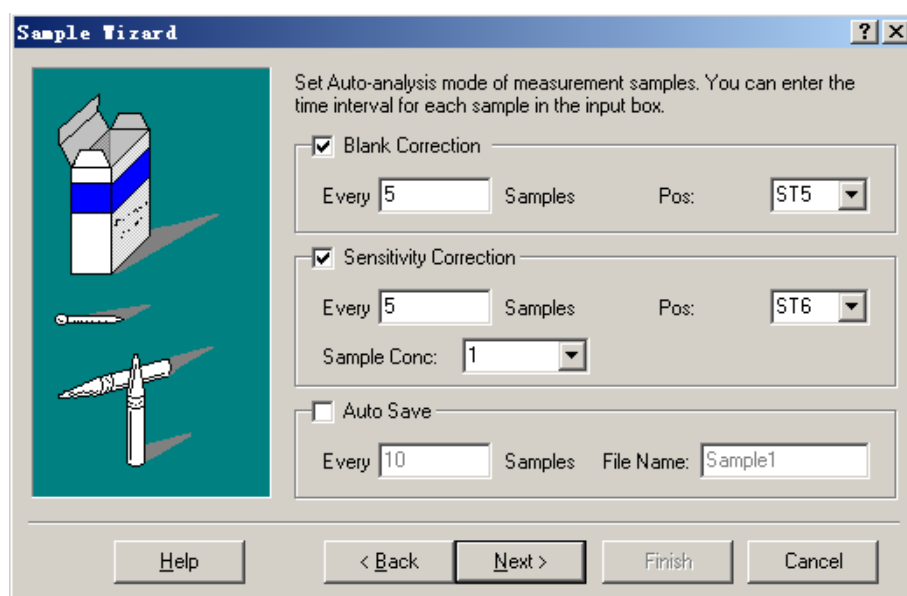

Fig. 3-3 Setting auto-analysis mode

You can set some auto-analysis functions in this page:
● Blank correction: set the period of running blank correction. If you set "every

5 samples", it will perform blank correction after measuring five samples. For this function, you should specify the right position of Blank Solution.

● Sensitivity correction: Similar as Blank correction, set the period of running sensitivity correction. You can input the concentration of correction standard in the "Sample Conc.". When measuring the standard, the system will calculate the sensitivity correction factor automatically, which will be used in the calculation of actual concentrations for later measurements. The formula of Sensitivity Correction Factor is as following:

$$\text{Sensitivity Correction Factor} = \frac{\text{Measured Conc.}}{\text{Set Conc.}}$$

For this function, you should specify the right position of Standard Solution for Sensitivity Correction.

• Auto Save: set to perform the auto-save the measured files periodically. You can input file name. The default file is "autosave.amd".

After you finish the setting, click "Next" to enter into the last window - settings for unknown samples, see Fig. 3-4.

Fig. 3-4 Setting the unknown samples

1. Sample counts: set the number of measuring samples (the standards are not included).

2. Sample name: input the sample name, such as "UNK-", which is a common name for all samples. If you want to specify an individual name for each sample, please refer to the instruction manual of AAWIN

❖

software.

3. Start ID: set the start number for a postfix after sample name.

4. Start Position: the default position for unknown samples is "S1". The user can also set any position as the start position, and then put samples in sequence. For example, set the sample counts as "20" and the start position "S20". The software will set the sample position as S20-S39 automatically. The user should put the samples in the corresponding positions.

5. The factors for Calculation of sample actual concentration. The formula of calculating the actual concentration is as follows:

$$Actual\,Conc. = \frac{Conc. \times VF \times DF \times CF \times SF}{WF}$$

VF refers to volume factor; DF refers to dilution factor; CF refers to correction factor; SF refers to sensitivity factor; WF refers to weight factor.

"Concentration" is calculated according to the ABS value. "VF", "DF", "CF" and "WF" are calculated according to your settings. "SF" is also calculated according to the formula we mentioned above. You can input the corresponding factors according to the specific samples. The default value is 1. If you do not set the factors, the actual concentration is equal to concentration after measurement.

If you need to modify any of all settings above, click on the button of "Back". Click on the button of "Finish" after you confirm all settings.

3.2 Setting parameters for measurement

To make a measurement, you need to set the measurement parameters first. Select [Settings] — [Measurement parameters] from the menu or click on the button ⏚ from the toolbar, and you will open a dialog box for measurement parameter settings. From the dialog box you will see four optional pages, the first page allows you to make regular settings. See Figure 3-5.

❖

Fig, 3-5 Setting regular parameters

You may set duplicate numbers for repeat measure of the "Standard", "Unknown", "Blank" and the "Sensitivity correction" in the "number of repeats" section. The range is 1 to 20.

After finishing setting the regular parameters, enter into the "Display" page to set the display of graphics.
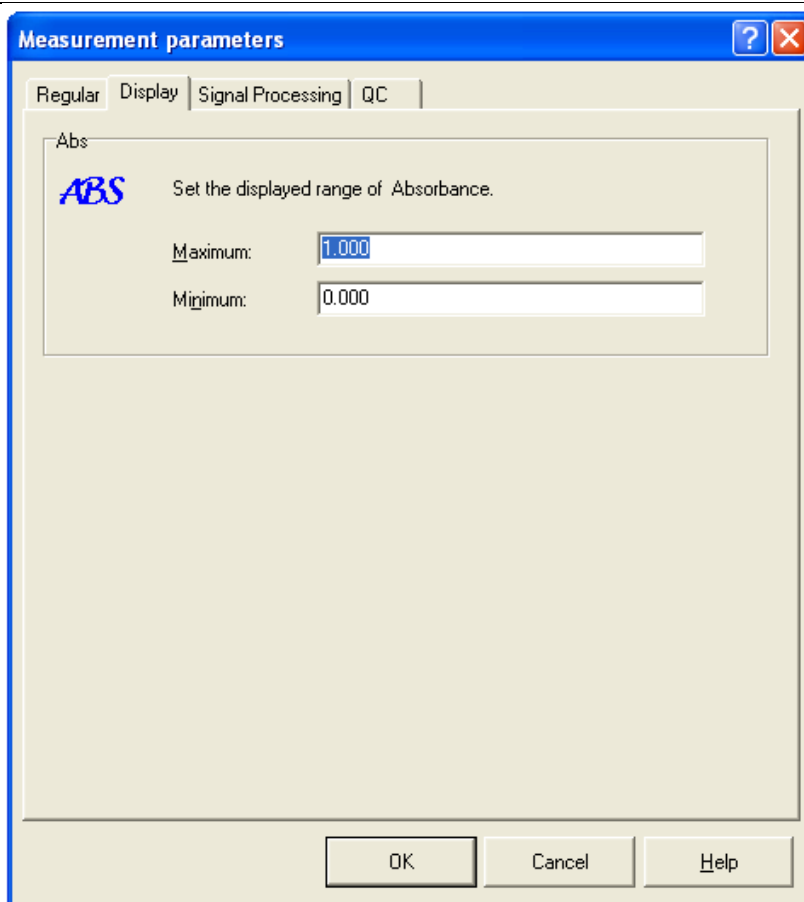
❖

Fig. 3-6 Setting the display

You can set the display range of absorbance in the measuring graphics, such as the lower and upper limits of the vertical axis of measuring graphics.

After finishing setting, you can enter into the "Signal processing" page to set parameters of signal processing, see Figure 3-7. In this page, you can select the calculation mode: Height or Area. You can input the integrating time. As the integrating time is set by the software automatically according to the heat procedure, so it can be neglected. If you need to modify the integrating time, it should be more than the calculated value. "Filter Factor" is a constant which needs to be given to filter the acquired data, i.e. the flatten factor. For the graphite furnace analysis, the value must be "0.1".
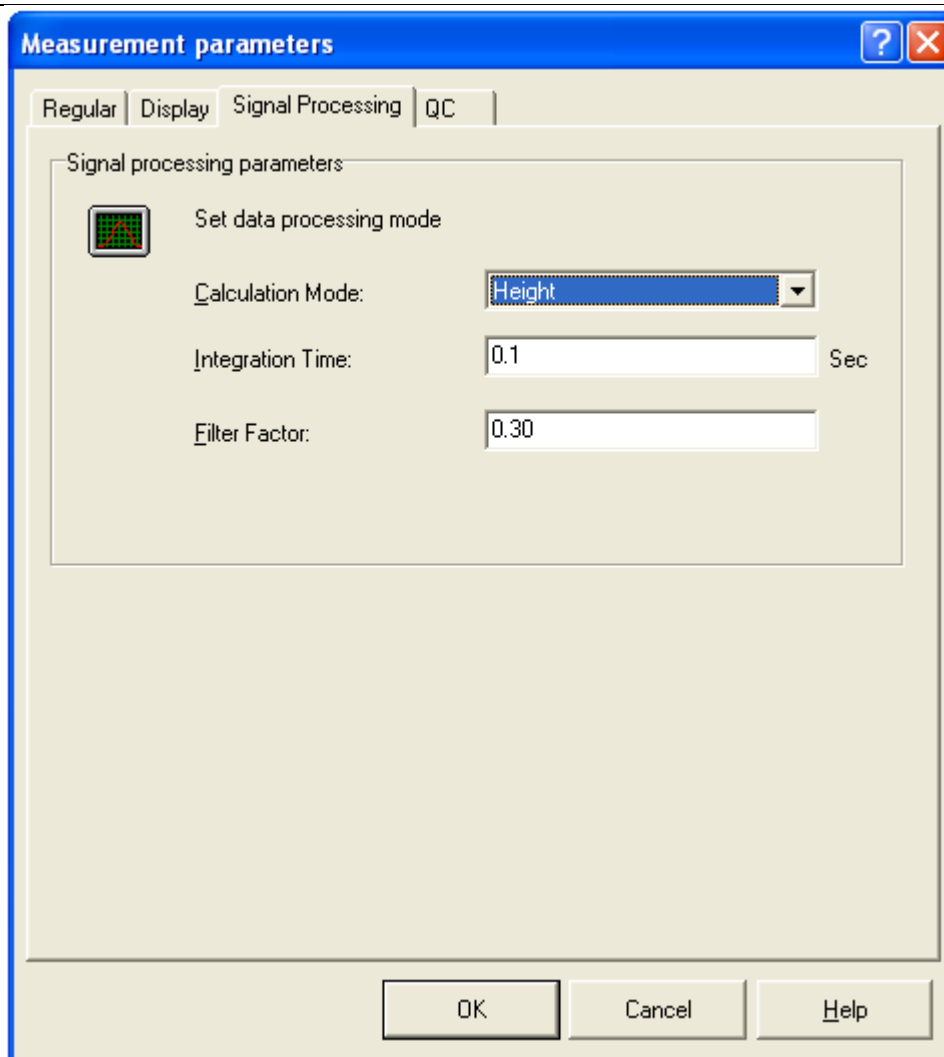
Figure 3-7 Signal processing

For better use of the Autosample, the function of Quality control is accompanied. You can make control of measured results and set the control options. See Figure 3-8.

- High value (upper limit) and low value (lower limit);
  Set the upper limit and lower limit of the quality control.
- High action and low action：
  Set the treatment when the measured value exceeds the limits. "Continue", "Stop" and "Remeasure" are available.

"Continue" means the QC inspects the unqualified products but the instrument continues measuring without any treatment.

"Stop" means the QC inspects the unqualified products, and the instrument stopped, waiting for the user's intervention.

"Remeasure" means the QC inspects the unqualified products, and the instrument performs remeasuring.

- Second action:

Set the treatment when the measured value exceeds the limits for the second time. The available methods are "Stop" and "Continue".
- Effective range: set the effective ABS range for which the QC is activated. That is, the quality control function is only used in the set ABS range if you choose and set the Effective range.
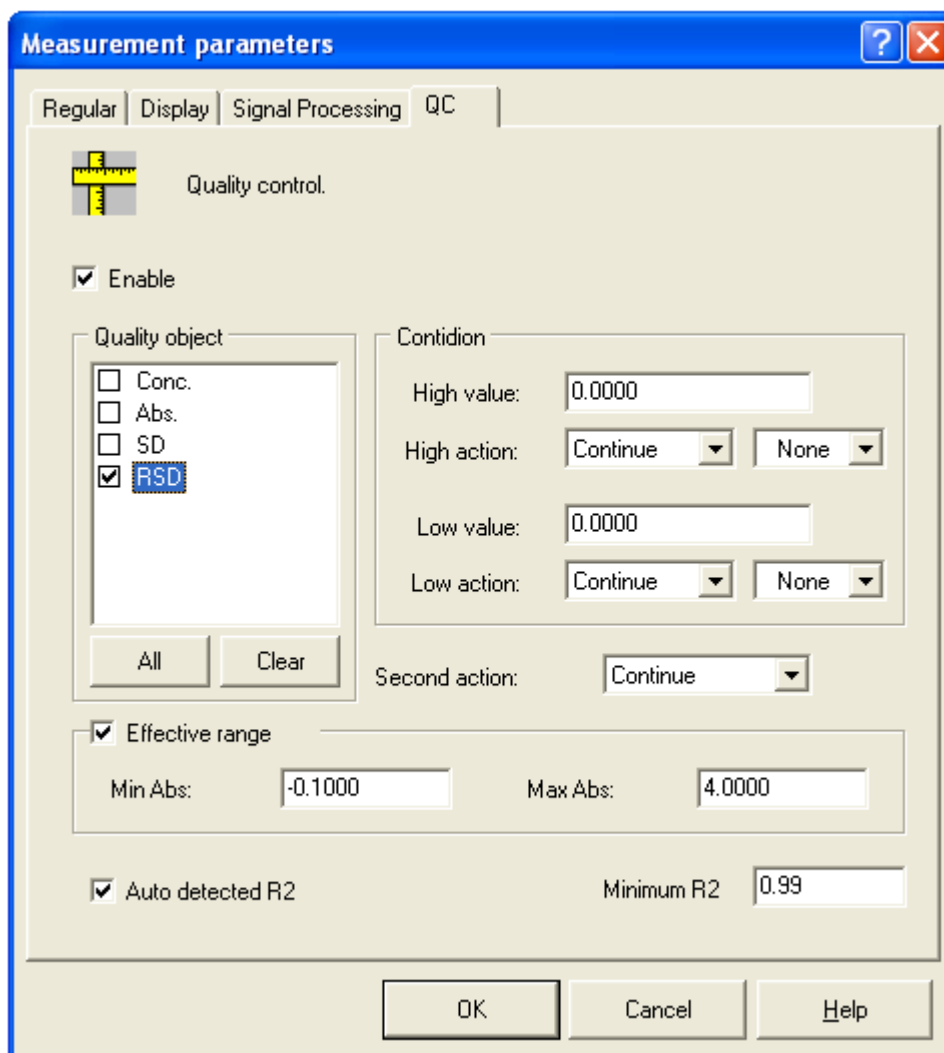

Fig. 3-8 Quality control

After finish setting, click "OK" to save and close the parameters setting dialog box.

3.3 Setting the temperature control

Click the "Heat" on the toolbar to enter into the setting page, see 3-9.

You should input the right heat procedures in this page, including temperature, elevation time, hold time, atomization and internal gas flow. Click "OK" to exit.

Fig.3-9 Setting the temperature control

3.4 Setting the sample position

Before measuring, the user can set any position as the actual sample position.

The AAWIN software will set the sequence positions for standards and unknown samples automatically according to "3.1 Setting sample wizard". The user can place samples according to the position information preset in the wizard. The user also can change the settings.

No.	Measure Type	Sample ID	Abs	Conc []	Actual Conc []	SD	RSD [%]	Pos	Date	Time
1	Blank							ST5		
2	Standard	STD -1		0.000				ST1		
3	Standard	STD -2		1.000				ST2		
4	Standard	STD -3		2.000				ST3		
5	Standard	STD -4		3.000				ST4		
6	Blank							ST5		
7	Sample	UNK -1						S		
8	Sample	UNK -2						S		
9	Sample	UNK -3						S		
10	Sample	UNK -4						S		
11	Sample	UNK -5						S		
12	Blank							ST		
13	Sensitivity correction			1.000				ST		
14	Sample	UNK -6						S		
15	Sample	UNK -7						S		
16	Sample	UNK -8						S8		
17	Sample	UNK -9						S9		

Edit...
Copy   Ctrl+C
Append   Ins
Delete   Del
Remeasure...
Sample Attribs...
Table Items...

Fig. 3-10 Setting the sample position

- See Fig.3-10: click on the preset position number under the column of "Pos."

❖

and make the position number is selected.

- Click your right mouse button to open a quick menu, and select the function of "edit".
- Edit the sample information and reset the position number.
- Click "OK" to exit.

3.5 Start measurement

After ensuring all the settings are correct, put the samples in the corresponding position on the tray.

Turn on the power of the graphite furnace; open the switch of cooling water, the regulator of argon gas cylinder and the ball valve switch of argon gas.

1．Empty burning. Before measuring, no less than twice empty burning of graphite tube are needed. Click "burn".
2．Wash. Click "wash" no less than twice to clean the capillary of autosampler.
3．Click "measure" to open the measure window. Then click the button of "Start". See Fig.3-11.
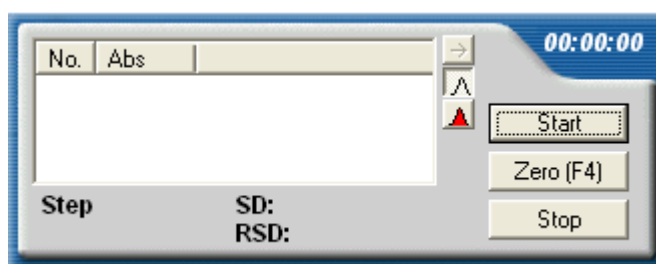

Fig 3-11 measuring page

4．Acquire the measuring results after measurement.
5．Process the measuring results.

Note: Do not make the instrument and the autosampler collided strongly while measuring. Do not try to hold up the moving mechanical parts.

The unexpected stop of power, cooling water and argon gas will stop the operation of the device. So please keep the continuous supply of power, cooling water and argon gas.

❖

Chapter 4 Replacing graphite tubes

1．Stop all operations of sampling.
2．Loosen the three fixing screw nuts which are used to fasten the autosampler onto the shoe plate, but do not remove them.
3．Pull the autosampler out to the utmost.
4．Click "graphite tube" on the toolbar to open the graphite furnace body.
5．Replace the graphite tube. Please notice to keep the light energy value constant before and after replacing the graphite tube.
6．Push the autosampler back to its original position.
7．Fasten the three fixing screw nuts on the shoes plate.
8．Repeat Strictly the steps of 19-21 in the "2.2 installation and adjustment".