



# **ZEE nit 700**



## 操作规程



德国耶拿分析仪器股份公司

# AAS Zeenit 700 原子吸收光谱仪

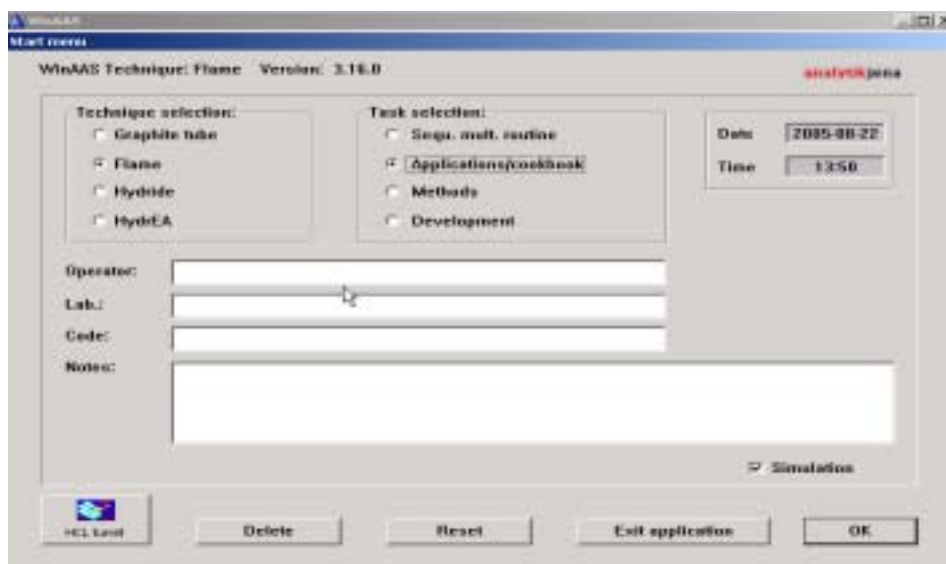
## 操作规程

### 火焰模式

#### 一、 开机

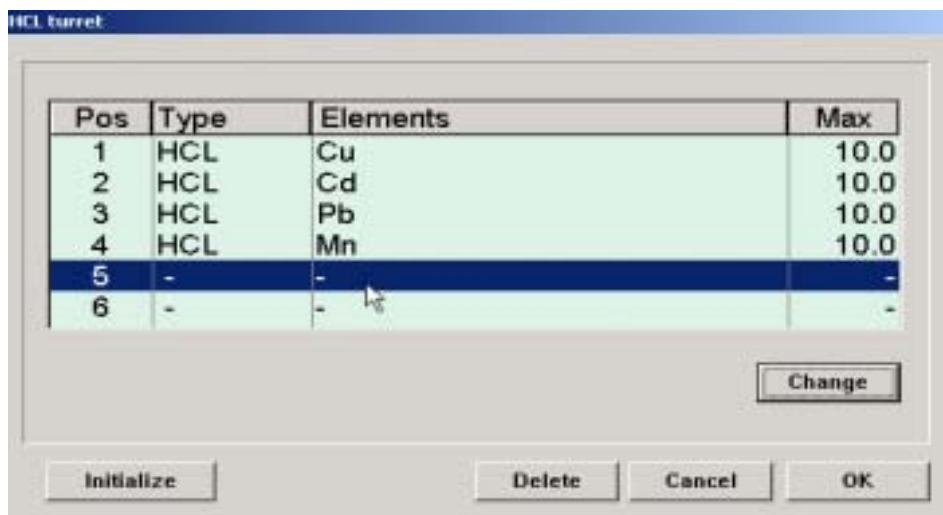
- 1、打开乙炔气瓶总阀，调节气体减压阀使气体出口压力为 0.1-0.15Mpa 左右。
- 2、打开空气压缩机电源，调节气体出口压力为 0.5 MPa 左右。
- 3、打开 Zeenit700 主机电源。
- 4、打开计算机电源。

- 5、双待仪器自检完毕后，双击 AAS 图标，进入应用软件的开始菜单界面：



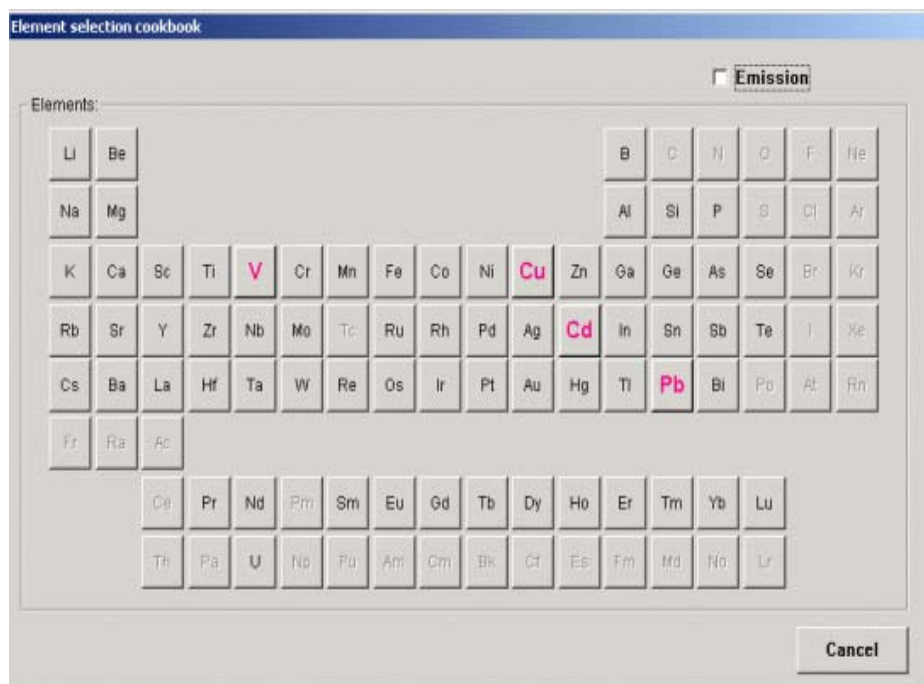
#### 元素空心阴极灯的安装：

点击  进入灯座菜单，在灯座相应位置安装元素空心阴极灯。

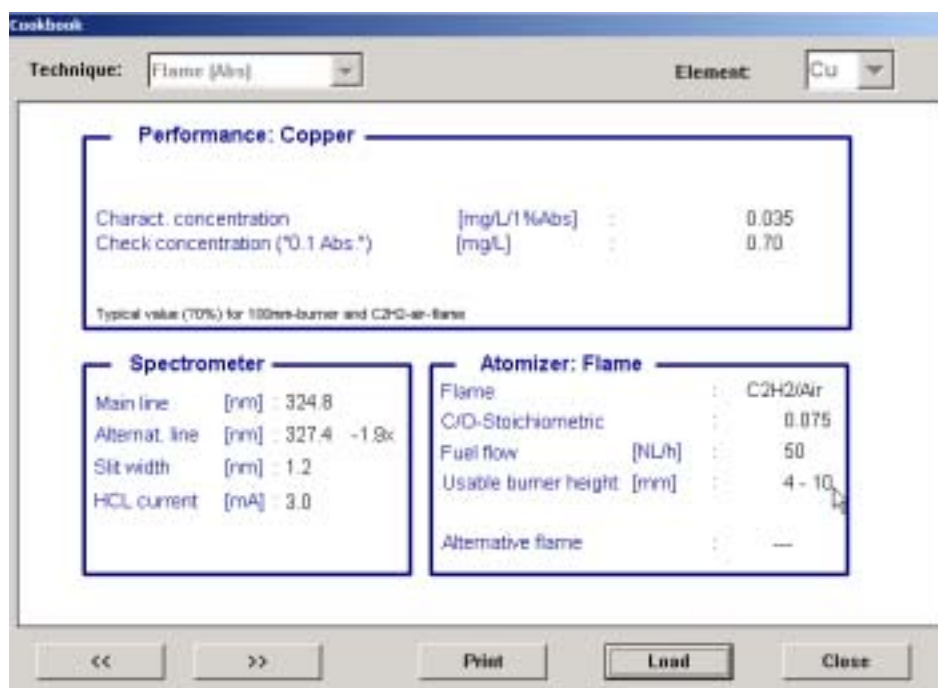


将光标移动到相应位置，点击 **Change** 或双击，将出现元素周期表，选择你要安装的空心阴极灯所对应的元素，最后点 OK 确认。

6、a. 选择 **Applications/Cookbook**( 厂家推荐参数 ),点击 OK ,出现以下界面：

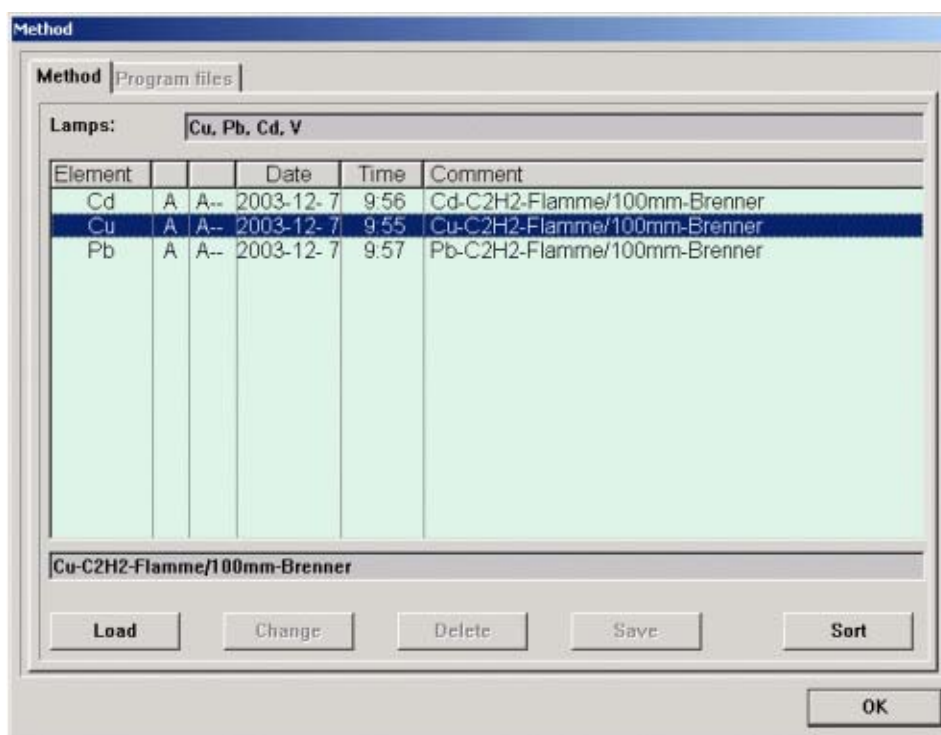


7、a. 选择待测元素 Cu，点击 **Cu** 按钮，进入该元素的 Cookbook 界面（厂家推荐参数）。

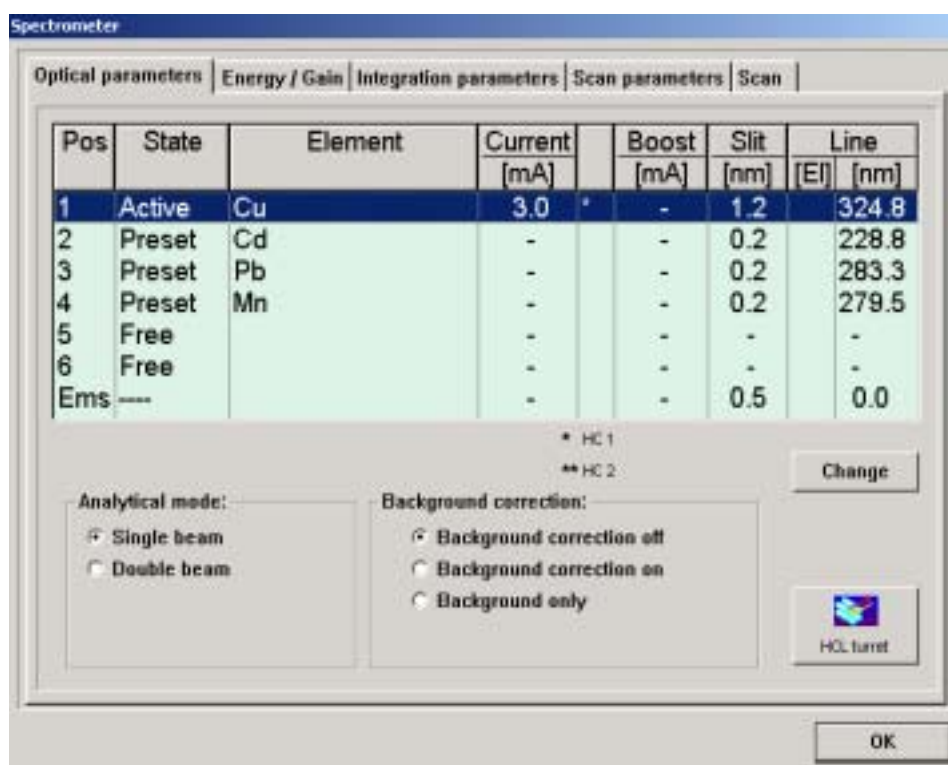


点击 Load 命令，进入初始化过程。

b.选择 **Method**(已存方法),点击 OK，进入下一个界面。

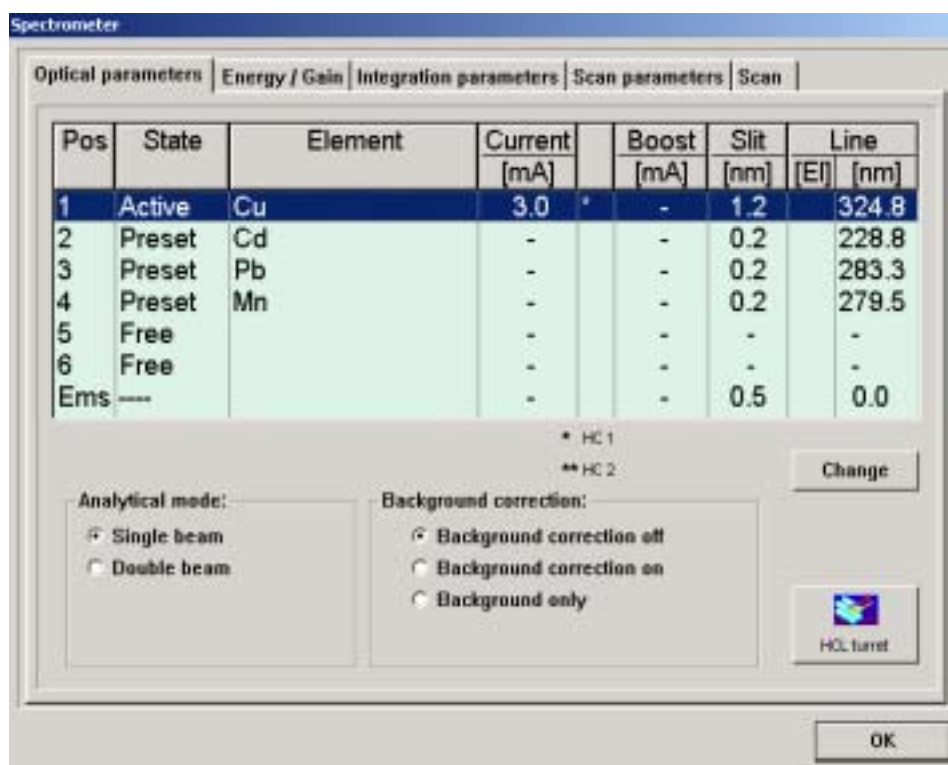


点击待测元素 Cu 所对应的方法，点击 Load 命令，进入初始化过程。  
8、初始化之后，点击“Spectrometer”，出现以下界面：

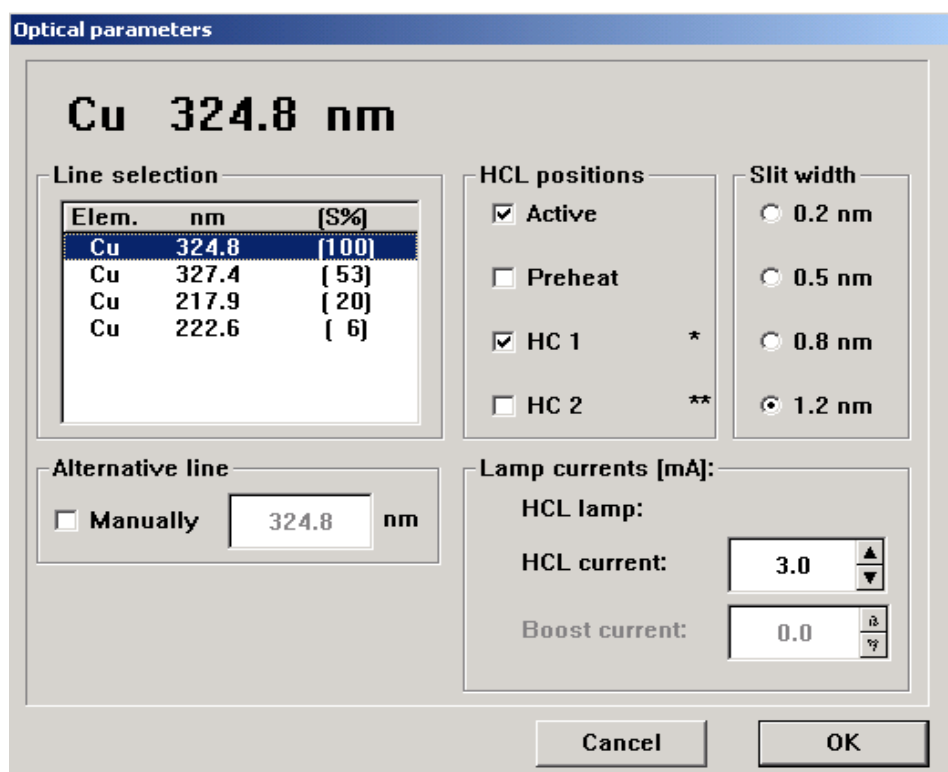


在“Spectrometer”界面内有五个标签：光学参数（Optical parameters），能量/增益（Energy/Gain），积分参数（Integration parameters），扫描参数（Scan parameters），扫描（Scan）。

(1) 点击“Optical parameters”按钮，出现以下界面：

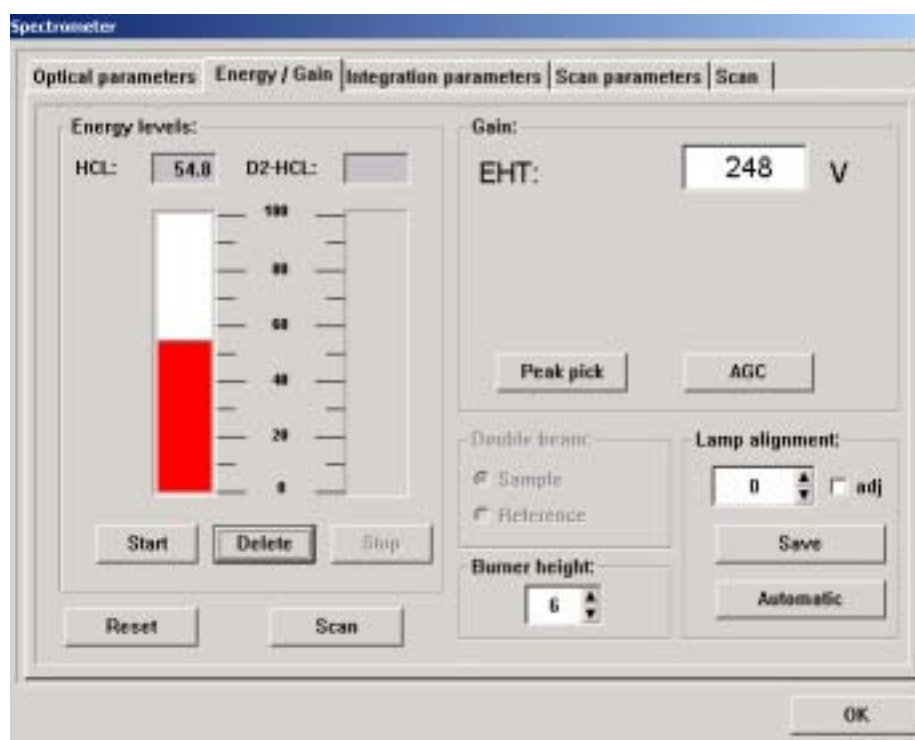


在这个界面里，点击 **Change** 按钮，进入光学参数界面：



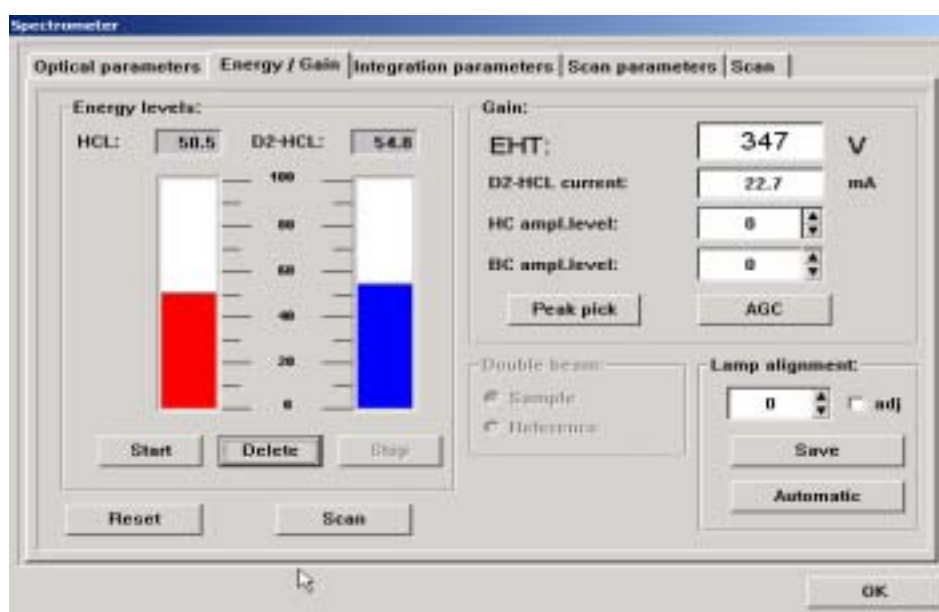
在光学参数界面内，可以选择或改变：元素分析线、灯的激活 Active 和预热状态 Preheat、狭缝宽度 slit、灯电流（调用 Cookbook 的参数时，如果使用的空心阴极灯是国产的，通常需要改变灯电流）。

(2)点击 “ Energy/Gain ” 按钮，出现以下界面：



在 **Lamp alignment** 里，点击 **Automatic** 按钮，仪器开始自动调节待测元素空心阴极灯光源能量至最佳状态，最后点击自动增益控制 **AGC** 按钮，结束能量最佳化过程。

**注：当选择氩空心阴极灯自动背景扣除时，待测元素空心阴极灯光源能量与氩空心阴极灯光源能量的比例关系调节请参照如下所述：**



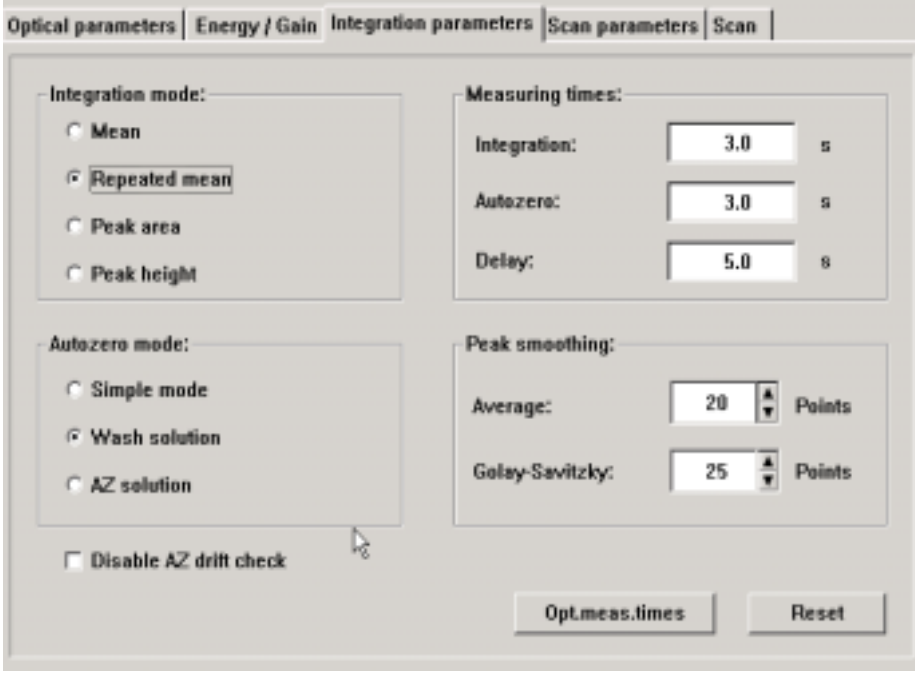
在 **Lamp alignment** 里，点击 **Automatic** 按钮，仪器开始自动调节待测元素空心阴极灯光源能量至最佳状态，改变 **HC ampl.level** 或 **BC ampl.level** 值（从 0 - 4），并点击自动增益控制 **AGC** 按钮，使

- a、空心阴极灯光源能量和氘空心阴极灯光源能量相差不超过 10%。
- b、氘空心阴极灯 (D2HCL Current) 电流在 10 - 30mA 之间。
- c、光电倍增管负高压 (EHT) 的值在 200 - 500 之间。

满足上述三个条件后，结束能量最佳化过程。

注：每改变一次 HC ampl.level 或 BCampl.level 值 (从 0 - 4)，便需点击自动增益控制  按钮，观察上述三个条件是否满足要求。

(3) 点击 “Integration parameters” 按钮，出现以下界面：



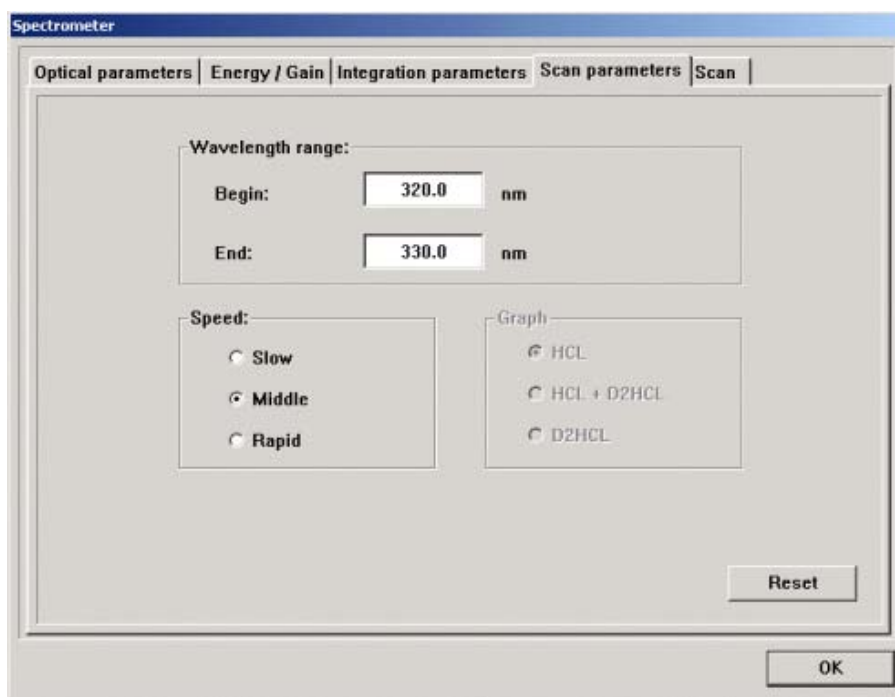
The image shows a software window titled "Integration parameters" with several tabs: "Optical parameters", "Energy / Gain", "Integration parameters" (selected), "Scan parameters", and "Scan". The window contains four main sections:

- Integration mode:** Radio buttons for "Mean", "Repeated mean" (selected), "Peak area", and "Peak height".
- Measuring times:** Input fields for "Integration:" (3.0 s), "Autozero:" (3.0 s), and "Delay:" (5.0 s).
- Autozero mode:** Radio buttons for "Simple mode", "Wash solution" (selected), and "AZ solution". A checkbox "Disable AZ drift check" is at the bottom.
- Peak smoothing:** Spinners for "Average:" (20 Points) and "Golay-Savitzky:" (25 Points).

At the bottom right, there are two buttons: "Opt.meas.times" and "Reset".

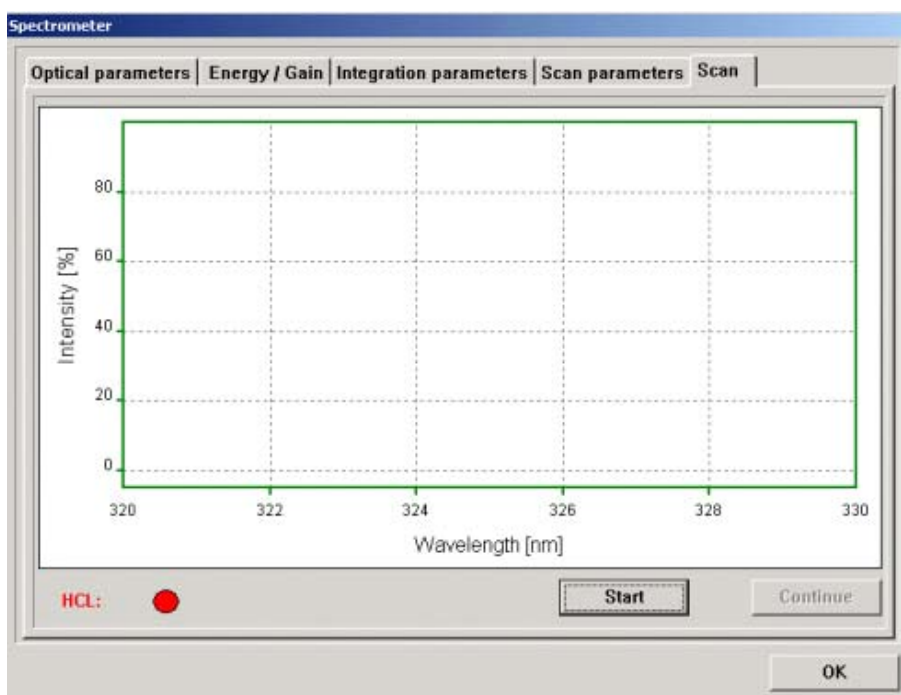
在积分参数界面，可根据以上数据设定。

(4) 点击 “Scan parameters” 按钮，出现以下界面：



在扫描参数界面里，输入扫描波长范围。输完之后，点击 SCAN 按钮进行波长扫描（此步可略）。

(5) 点击 “Scan” 按钮，出现以下界面：



在扫描界面里，点击 START 按钮，开始波长扫描，扫描结束后，显示分析线波长（此步可略）。

最后点击 OK 键，退出 “Spectrometer” 界面。

**注：最后两步一般只在质量鉴定时才使用，测试时狭缝宽度 slit 应选为 0.2nm。**

9、点击“Flame”，进入火焰和气体控制界面：

Flame and gas control

Control | Flame optimization | Parameters | Automatic flame optimization

Flame state: Ready Burner: 100 mm

Sensors: Pressures: Gas flow:

Flame: Off Fuel: - Fuel: l/h

Siphon: OK Nebulizer: Oxidant: l/h

Waste bottle: OK Air: OK Tot.Ox.: l/h

N2O: OK Gas/Ox: l/h

Test air Test N2O Test fuel Test end

Ignite flame Air -> N2O Extinguish flame

OK

点在 Flame 界面里，先后点击“Test Air”和“Test Fuel”进入以下界面：

Flame and gas control

Control | Flame optimization | Parameters | Automatic flame optimization

Flame state: Check Burner: 100 mm

Sensors: Pressures: Gas flow:

Flame: Off Fuel: OK Fuel: 50 l/h

Siphon: OK Nebulizer: 1.2 Oxidant: 403 l/h

Waste bottle: OK Air: OK Tot.Ox.: 403 l/h

N2O: OK Gas/Ox: 0.124

Test air Test N2O Test fuel Test end

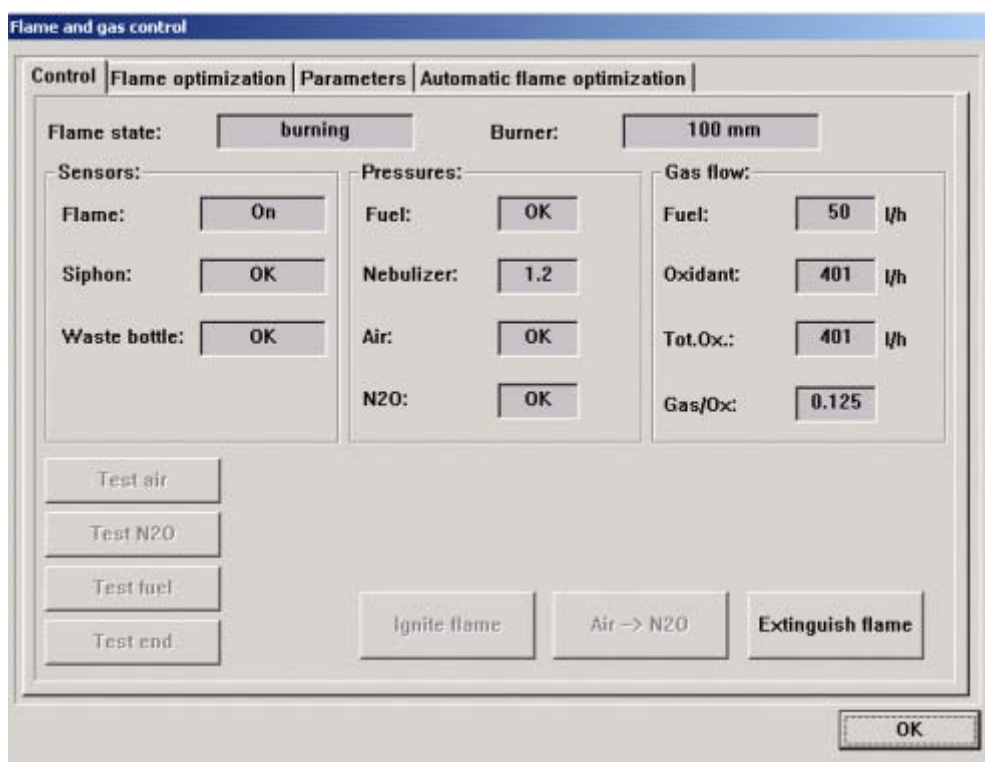
Remaining for test: 11 s

Ignite flame Air -> N2O Extinguish flame

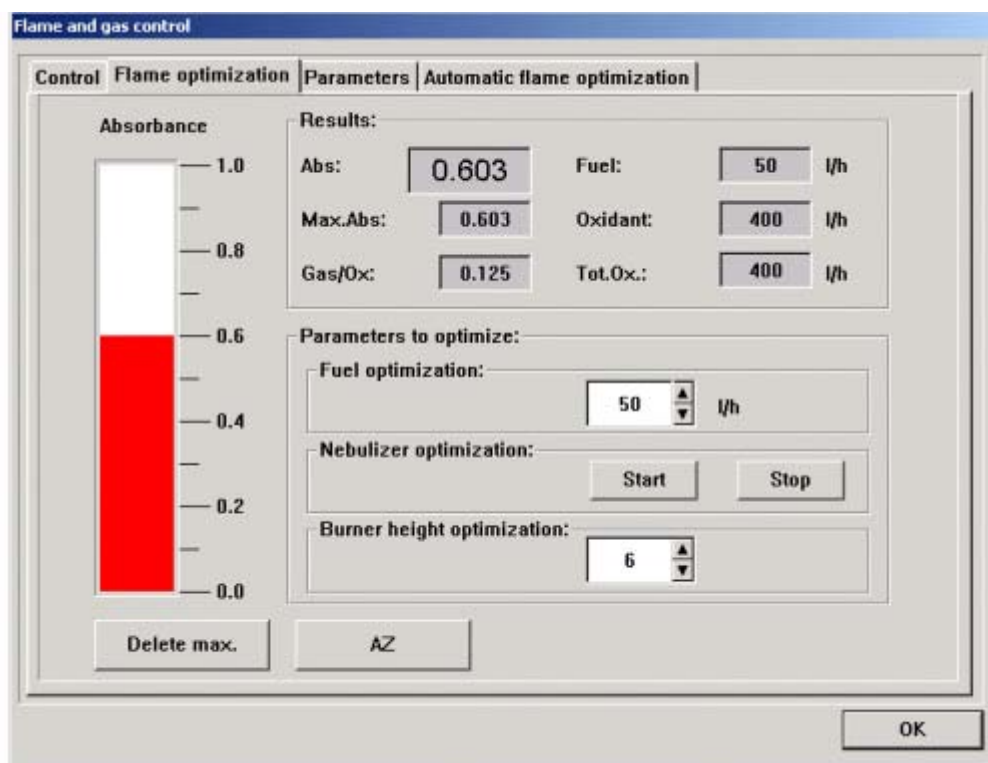
OK

检测空气和燃气流量和压力，当出现类似以上状态时，点击“Test end”结束检测过程。

接着，点击“Ignite”点燃火焰。如下所示：



- 10、 点燃火焰后，点击“**Flame Optimization**”，用空白液校零（AZ），用 1.0ppmCu 标准溶液调节燃烧头-雾化系统的最佳雾化效率，调节雾化器及撞击球的位置，使吸光度值达最大值。（如雾化效率已最佳化，此步可略。**注意：此步仅在清洗燃烧头-雾化系统后才需最佳化**）。火焰优化界面如下：



最后点击 OK 键，退出“Flame”界面。

- 11、 点击“Autosampler”，进入自动进样器的编辑界面（如果用户没有选配自动进样器，此步可略）。

The screenshot shows the 'Autosampler' configuration window with several tabs: 'Autosample mode', 'Positions', 'Techn. parameters', 'Dilution/IB', 'Function tests', and 'Error check'. The 'Autosample mode' tab is active. It contains three main sections: 'Autosampler type' with radio buttons for 'No autosampler', 'AS51', 'AS52', 'AS52 S', 'MPE50', and 'MPE60'; 'Tray type' with radio buttons for '89 Positions', '53 Positions', and '50 Positions'; and 'Working mode' with radio buttons for 'Step [1 cycle]' and 'Continuous'. The 'Washing' section has radio buttons for 'Off', 'Between samples', 'Between measurements', and 'Between components', along with input fields for 'Wash cycles' and 'Wash time [s]'. The 'Injection switch' section has radio buttons for 'Off' and 'Activate SFS 5', and input fields for 'Load time' and 'Injection time'. The 'Sample volume' section has input fields for 'Injection volume' and 'Dip time'. A 'Reset' button is located at the bottom right of the main configuration area, and an 'OK' button is at the bottom right of the window.

- 12、 标样测量 - 制作标准曲线

(1) 点击“Calibration”，选择标准校正模式（Standard calibration），进入校正界面：

The screenshot shows the 'Calibration' window with tabs: 'Calibration mode', 'Conditions', 'Statistics', 'Conc. input', 'Table', 'Curve parameters', and 'Recalibration'. The 'Calibration mode' tab is active. It contains a section titled 'Computation / calibration method:' with radio buttons for 'Absorbance mode (no calibration)', 'Standard calibration', 'Bracketing calibration', 'Method of addition', and 'Method of Additions calibration'. A 'Reset' button is located at the bottom right of the main configuration area, and an 'OK' button is at the bottom right of the window.

在校正界面内，有校正模式(Standard mode)、条件设定(Conditions)、数学统计(Statistics)、标样浓度输入(Conc. Input)、标样运行表格(Table)、曲线参数(Curve parameters)。

(2) 点击“Conditions”，进入如下界面：

The screenshot shows the 'Conditions' tab of the Calibration software. The interface includes several sections for setting up the calibration process:

- Standards:**
  - Making standards:** Radio buttons for 'Using premixed standards' (selected), 'By different volumes', and 'Mixed by autosampler'.
  - No. of standards:** A numeric input field set to 5.
  - Sample meas. without add:** A numeric input field set to 3.
- Blank correction:** Radio buttons for 'Absorbance corrected' (selected) and 'Concentration corrected'.
- Other options:** Checkboxes for 'Continue after curve calculation' and 'AZ (AZ/AM) between standards'.
- Getting values by:** Radio buttons for 'Measurement' (selected) and 'Manual input'.
- Mix conditions:**
  - Filling level mix cup:** A numeric input field set to 5 ml.
  - [sample part]:** A numeric input field set to 2.5 ml.
  - Sample volume:** A numeric input field set to 0 ml.
  - Max. add. volume:** A numeric input field set to 10 ml.
  - Total modifier vol.:** A numeric input field set to 0 ml.

Buttons for 'Reset', 'OK', and 'Recalibration' are visible at the bottom of the window.

在条件设定界面，可根据以上数据和选项设定。标准数量 No. of standards 须根据实际情况而定。

(3) 点击“Statistics”，进入如下界面：

The screenshot shows the 'Statistics' tab of the Calibration software. The interface includes several sections for setting up statistical analysis:

- Statistics:** Radio buttons for 'Off', 'Sigma statistics' (selected), and 'Median Statistics'.
- Grubbs outlier test:** Radio buttons for 'Off' (selected), 'On (Flagging mean by "[I]")', and 'On [Without flagging]'.
- Measurement cycles:**
  - Runs/Std.:** A numeric input field set to 3.
  - Blind runs/Std.:** A numeric input field set to 1.
- Statistical output:** Checkboxes for 'Standard deviation [SD]' and 'Relat. standard deviation [RSD]' (both selected).

Buttons for 'Reset' and 'OK' are visible at the bottom of the window.

在数学统计界面，可根据以上数据和选项设定。

(4) 点击 “Conc. Input ”, 进入如下界面：

**Calibration**

Calibration mode | Conditions | Statistics | **Conc. input** | Table | Curve parameters | Recalibration

**Stock standards**

Kind of stock standards:

☒ Liquid

☐ Solid

Concentrations:

|                                            | Conc.  |
|--------------------------------------------|--------|
| Stock solution 1:                          | 1.0000 |
| <input type="checkbox"/> Stock solution 2: | 1.0000 |
| <input type="checkbox"/> Stock solution 3: | 1.0000 |
| <input type="checkbox"/> Stock solution 4: | 1.0000 |

**Concentration unit:**

☒ mg/L

☐ user defined

☐ 微g/L

☐ 微g/mL

☐ ng/mL

☐ ng/L

Conv.factor:

1.00000000

**Absolute unit:**

☐ mg

☐ 微g

☒ ng

☐ pg

☐ fg

Reset

OK

在这个界面里，选择浓度单位即可,如（mg/L）。

(5) 点击 “Table ”, 进入如下界面：

**Calibration**

Calibration mode | Conditions | Statistics | Conc. input | **Table** | Curve parameters | Recalibration

| Standards: |          |       |     | Conc. | Mean |    |         |
|------------|----------|-------|-----|-------|------|----|---------|
| No         | Name     | State | Pos | mg/L  | Abs  | SD | RSD [%] |
| 1          | Cal-Zero | (-)   | ### | 0.000 |      |    |         |
| 2          | Cal-Std1 | (1)   | ### | 0.100 |      |    |         |
| 3          | Cal-Std2 | (1)   | ### | 0.250 |      |    |         |
| 4          | Cal-Std3 | (1)   | ### | 0.500 |      |    |         |
| 5          | Cal-Std4 | (1)   | ### | 0.750 |      |    |         |
| 6          | Cal-Std5 | (1)   | ### | 1.000 |      |    |         |

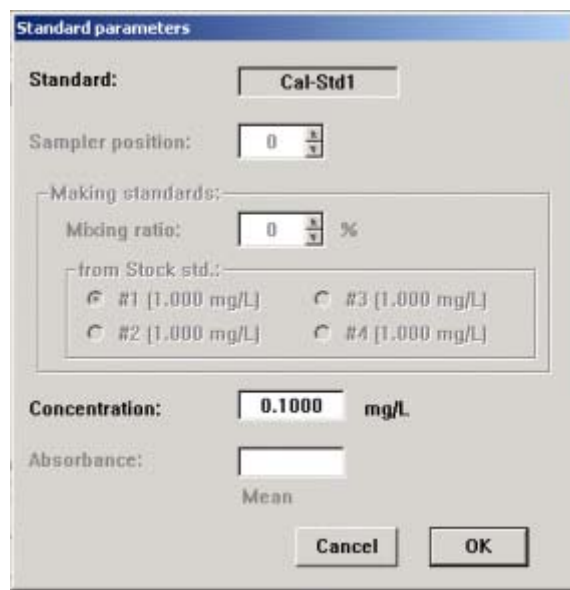
Clear Table Set recal Check Run sample

Delete line Parameters Fit curve -> Blindw.

OK

其中，Cal-Zero 为校正零点，即空白零点。

点击“Parameters”按钮或双击，进入标准系列浓度输入的对话框：

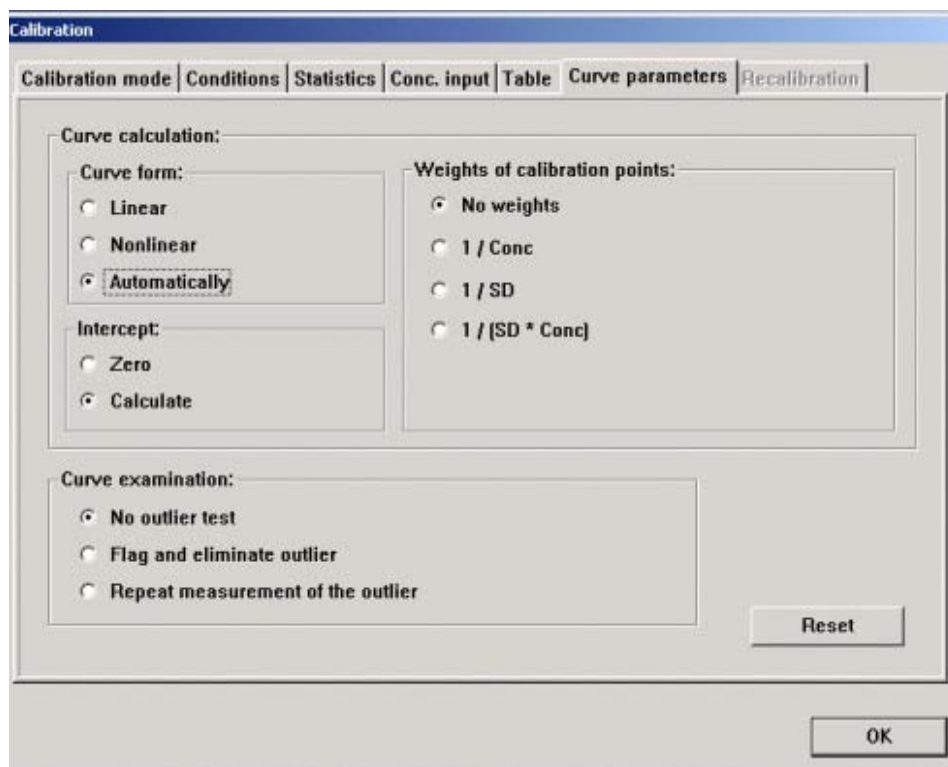


The "Standard parameters" dialog box contains the following fields and options:

- Standard:** A dropdown menu set to "Cal-Std1".
- Sampler position:** A text box containing "0" and a small unit icon.
- Making standards:** A section containing:
  - Mixing ratio:** A text box with "0" and a percentage sign.
  - from Stock std.:** A group box containing four radio buttons:
    - ☒ #1 (1,000 mg/L)
    - ☐ #2 (1,000 mg/L)
    - ☐ #3 (1,000 mg/L)
    - ☐ #4 (1,000 mg/L)
- Concentration:** A text box with "0.1000" and a unit "mg/L".
- Absorbance:** A text box with "Mean" below it.
- Buttons:** "Cancel" and "OK" at the bottom right.

依次输入标准样品的浓度，直到输完为止。

(6) 点击“Curve parameters”，进入如下界面：

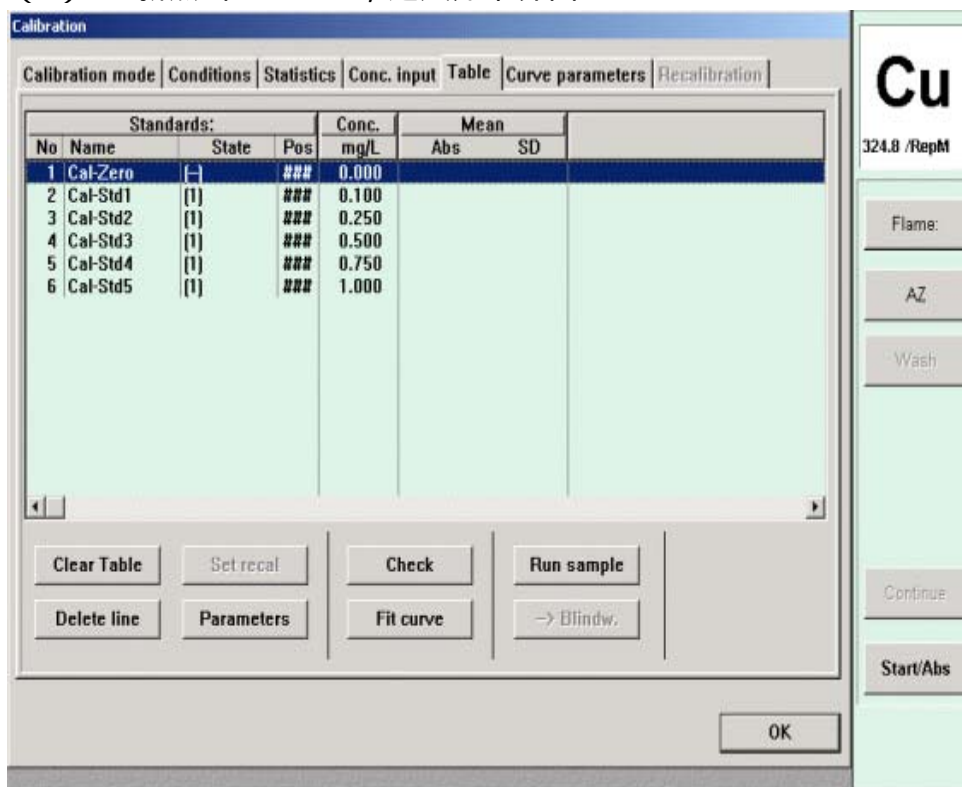


The "Calibration" dialog box has several tabs: "Calibration mode", "Conditions", "Statistics", "Conc. input", "Table", "Curve parameters" (selected), and "Recalibration". The "Curve parameters" tab contains the following sections:

- Curve calculation:**
  - Curve form:** Radio buttons for "Linear", "Nonlinear", and "Automatically" (selected).
  - Intercept:** Radio buttons for "Zero" and "Calculate" (selected).
- Weights of calibration points:** Radio buttons for "No weights" (selected), "1 / Conc", "1 / SD", and "1 / (SD \* Conc)".
- Curve examination:** Radio buttons for "No outlier test" (selected), "Flag and eliminate outlier", and "Repeat measurement of the outlier".
- Buttons:** "Reset" and "OK" at the bottom right.

在这个界面，可根据以上选项设定。

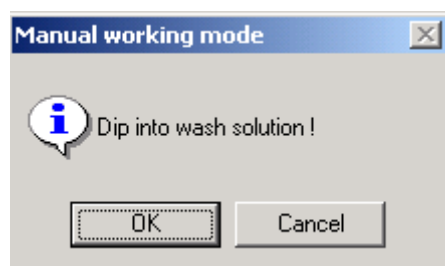
(7) 重新点击“Table”，进入如下界面：



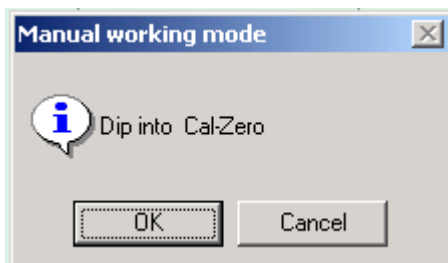
在这个界面里，点击“Start/Abs”，开始标样的测量。

接下来，会提示测量的信息：

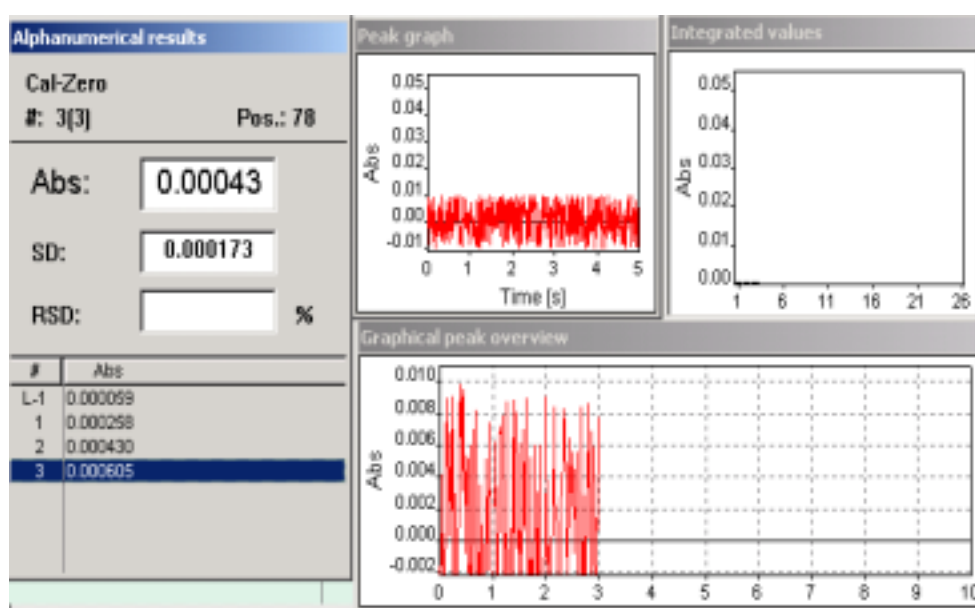
A、将吸样管放入清洗液中，点击 OK 进行自动校零。



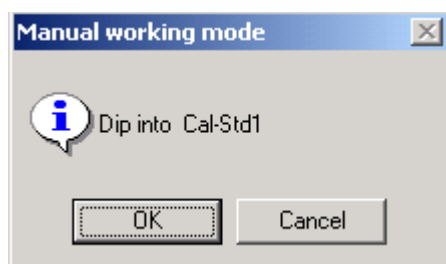
B、将吸样管放入空白溶液中，点击 OK，进行空白测量。



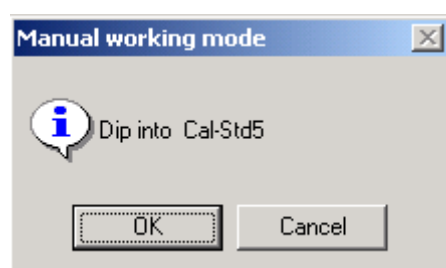
测量显示如下：



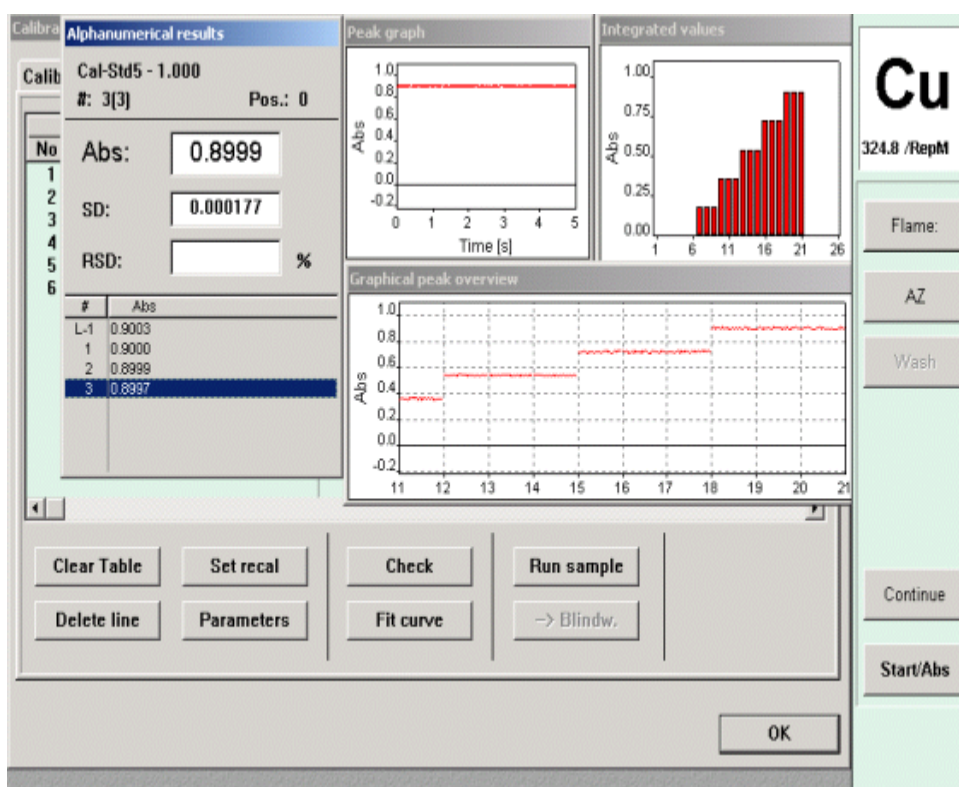
C、空白测量完毕之后，将吸样管放入标样 1，点击 OK，进行标样 1 测量。根据提示测量完所有的标样。



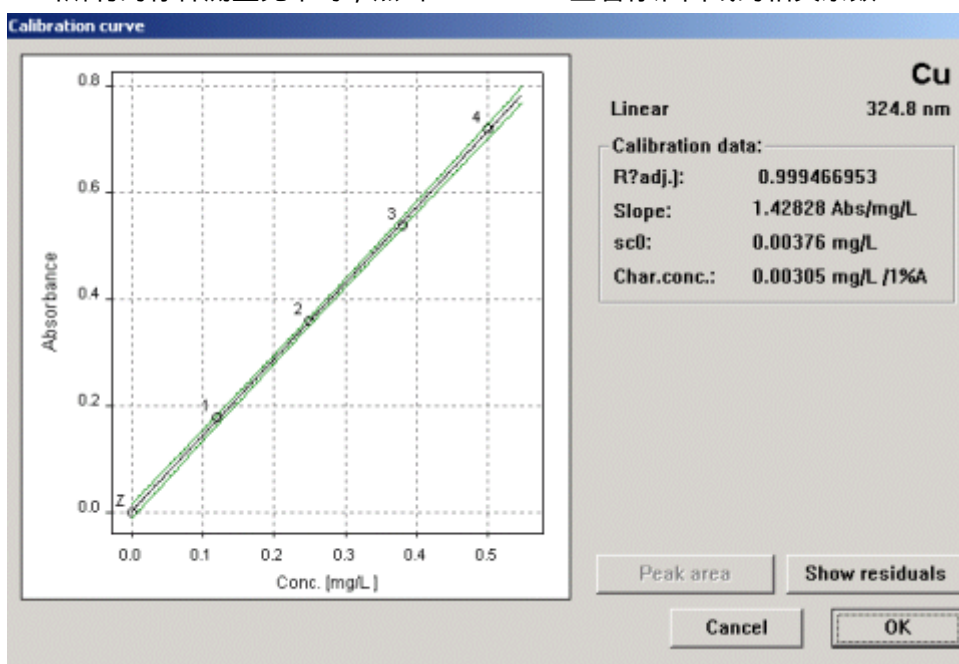
- o
- o
- o



测量完毕提示如下：



当所有的标样测量完毕时，点击“Fit curve”查看标准曲线的相关系数：



如果  $R^2 \geq 0.995$ ，标准曲线良好，可以进入待测样品测量。否则，需删除某个点或另配标样，点击“Run sample”，重新测量不准确的标样。

最后点击 OK 键，退出“Calibration”界面。

### 13、 样品测量。

(1) 点击“Samples”，进入样品测量界面：

**Samples**

Sample table | Conc. output | ID samples | Statistics | Mult.Param.

| No | * | Solution |     | ID/Wt-Data |        |          | Actions |       |      |
|----|---|----------|-----|------------|--------|----------|---------|-------|------|
|    |   | Type     | Pos | Name       | Pre-DF | Vol.[mL] | Wt[g]   | Cal.  | Tube |
| 1  | * | Sample   | ### |            | 1.000  |          |         | Rec/C |      |
| 2  | * | Sample   | ### |            | 1.000  |          |         |       |      |
| 3  | * | Sample   | ### |            | 1.000  |          |         |       |      |
| 4  | * | Sample   | ### |            | 1.000  |          |         |       |      |
| 5  | * | Sample   | ### |            | 1.000  |          |         |       |      |
| 6  |   | Sample   | ### |            | 1.000  |          |         |       |      |
| 7  |   | Sample   | ### |            | 1.000  |          |         |       |      |
| 8  |   | Sample   | ### |            | 1.000  |          |         |       |      |
| 9  |   | Sample   | ### |            | 1.000  |          |         |       |      |
| 10 |   | Sample   | ### |            | 1.000  |          |         |       |      |
| 11 |   | Sample   | ### |            | 1.000  |          |         |       |      |
| 12 |   | Sample   | ### |            | 1.000  |          |         |       |      |
| 13 |   | Sample   | ### |            | 1.000  |          |         |       |      |
| 14 |   | Sample   | ### |            | 1.000  |          |         |       |      |
| 15 |   | Sample   | ### |            | 1.000  |          |         |       |      |

Buttons: Init.Table, Change, Load / Save, Single values, Final actions (Lamp off, Flame off), Delete results, Delete, Working area, -> Cal.tab., Print table, Insert, Run sample, Show sequence, Recalculate, Export [CSV], Clipboard, OK

在这个界面，有样品运行表格(Sample table)、样品浓度输出(Conc.output)、样品名称输入(ID sample)、数学统计(Statistics)。

点击工作区“Working area”，设定要测量的待测样品数量如 5 个样品：

**Working area**

Table rows to be analyzed:

1-5

[f.e. 1-10,18,20-25]

Buttons: Cancel, OK

(2) 点击“Conc.output”，进入如下界面：

**Samples**

Sample table | Conc. output | ID samples | Statistics | Mult.Param.

Concentration output (Conc2) for:

☒ Liquid original sample

☐ Solid original sample

Output style:

Absorbances:

☐ 3 signif. digits

☒ 4 signif. digits

☐ 5 signif. digits

Concentrations:

☐ 3 signif. digits

☒ 4 signif. digits

☐ 5 signif. digits

Concentrations:

☒ mg/L

☐ 微/L

☐ 微/mL

☐ ng/mL

☐ ng/L

☒ user defined

Conv.factor:

Reset

OK

在这个界面，可根据以上选项设定。

(3) 点击“ID sample”，点击“Sample table → ID”，出现如下界面：

**Samples**

Sample table | Conc. output | ID samples | Statistics | Mult.Param.

Sample ID-Fixed part:

Batch:

Customer order:

Sample ID / Fixed part:

Solution of solid samples:

Sol. volume:  ml

Nominal weight:  g

| No | Pos | Name | Pre-DF | Vol.[mL] | Wt[g] | Blank corr |
|----|-----|------|--------|----------|-------|------------|
| 1  | ### |      | 1.000  |          |       | Off        |
| 2  | ### |      | 1.000  |          |       | Off        |
| 3  | ### |      | 1.000  |          |       | Off        |
| 4  | ### |      | 1.000  |          |       | Off        |
| 5  | ### |      | 1.000  |          |       | Off        |
| 6  | ### |      | 1.000  |          |       | Off        |
| 7  | ### |      | 1.000  |          |       | Off        |
| 8  | ### |      | 1.000  |          |       | Off        |
| 9  | ### |      | 1.000  |          |       | Off        |
| 10 | ### |      | 1.000  |          |       | Off        |
| 11 | ### |      | 1.000  |          |       | Off        |

Sample table → ID    Change line    Insert line    Save    → Sample table

Delete table    Delete    Append line    Load    Synopsis

OK

(4) 双击其中一行，出现如下界面：

**Sample parameters**

Table row number:

ID/Wt data:

Autosampler cup position:

Sample name:

Pre-dilution factor:

Sol. volume:  ml

Weight:

Relative humidity:  %

☐ Blank correction

在这个界面中，输入样品名称，如 WATER。

(5) 点击“Statistics”，进入如下界面：

**Samples**

Sample table | Conc. output | ID samples | **Statistics** | Mult. Param. |

Statistics:

☐ Off  
☒ Sigma statistics  
☐ Median Statistics

Cycles:

Measurement cycles:   
Runs/QC sample:   
Blind cycles:

Statistical output:

☒ Standard deviation (SD)  
☒ Relat. standard deviation (RSD)  
☐ Relative homogeneous factor (RHF)

Grubbs outlier test:

☒ Off  
☐ On (Flagging mean by "[I]")  
☐ On (Without flagging)

Confidence interval calculation:

☒ Off  
☐ Absolute  
☐ Relative

Confidence levels:

☐ 68.3 % (1 Sigma) ☐ 98 %  
☐ 90 % ☐ 99 %  
☐ 95 % ☐ 99.7 % (3 Sigma)  
☒ 95.4 % (2 Sigma) ☐ 99.9 %

在这个界面，可根据以上数据和选项设定。

(6) 重新点击“Samples”，进入样品测量界面：

**Samples**

Sample table | Conc. output | ID samples | Statistics | Mult.Param.

| No | * | Solution |     | ID/Wt.Data |        |          |       | Actions |      |    |
|----|---|----------|-----|------------|--------|----------|-------|---------|------|----|
|    |   | Type     | Pos | Name       | Pre-DF | Vol.[mL] | Wt[g] | Cal.    | Tube | AZ |
| 1  | * | Sample   | ### |            | 1.000  |          |       | Rec/C   |      |    |
| 2  | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 3  | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 4  | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 5  | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 6  | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 7  | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 8  | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 9  | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 10 | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 11 | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 12 | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 13 | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 14 | * | Sample   | ### |            | 1.000  |          |       |         |      |    |
| 15 | * | Sample   | ### |            | 1.000  |          |       |         |      |    |

Buttons: Init.Table, Change, Load / Save, Single values, Final actions, Delete results, Delete, Working area, -> Cal.tab., Lamp off, Flame off, Print table, Insert, Run sample, Show sequence, Recalculate, Export [CSV], Clipboard, OK

Right panel: **Cu**, 324.8 /Repm, Flame, AZ, Wash, Continue, Start/Conc

点击“ **Start/Conc** ”，连续测量或“ **Run sample** ”单个样品测量。测量界面类似标样测量。

**注意：**测量样品之前，可以用空白溶液，点击 **AZ** 按钮校零。

#### 14、 测试结果打印。

a)、样品测试完毕后，点击 **Print table**，进入如下界面：

**Print**

**Print options**

☒ **Concentration values**

☐ **Absorbance values**

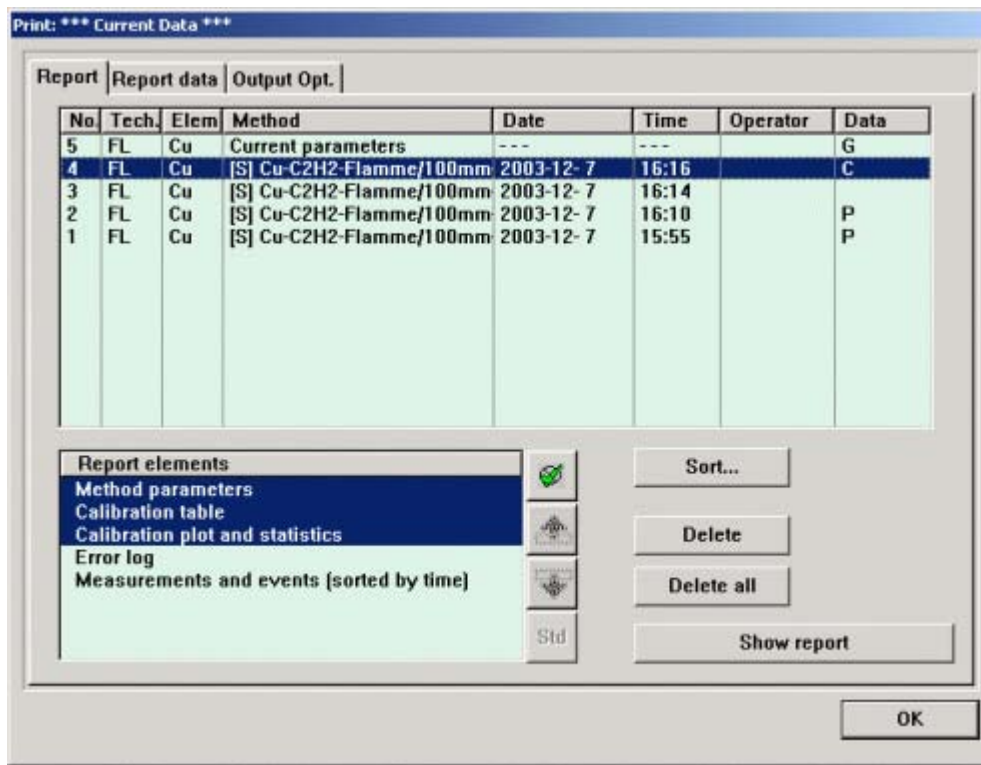
☐ **Absorbances and concentration values**

Buttons: Cancel, OK

选择打印吸光值或/和浓度值报表。

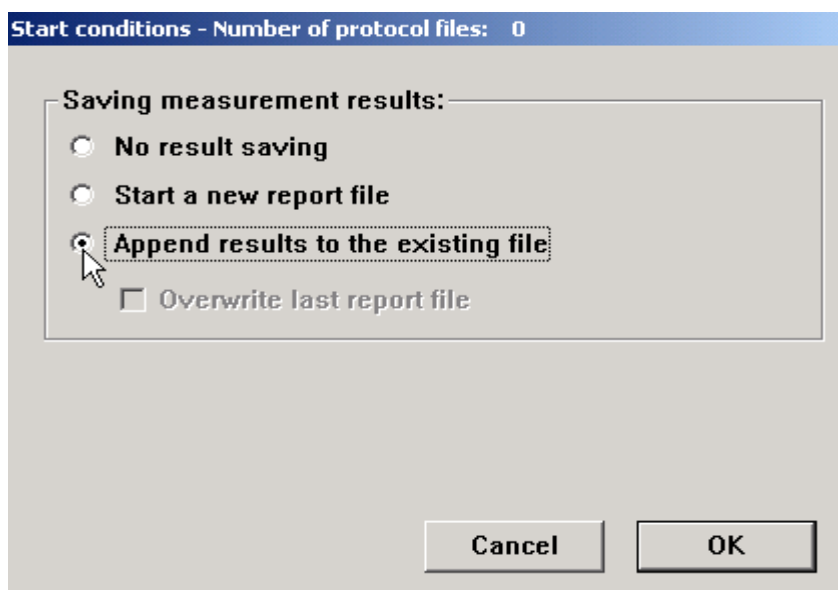
测试结果转存：点击 **CSV file**，可将结果转存到任何路径下保存，并可通过 Excel 文档对测试结果进行编辑。

2)、点击“Print”命令，



点击“Show report”，将显示结果报告。根据需要，选择是否打印或储存结果。

注：若需将测试结果与标准曲线打印在同一张纸上，做完标准曲线后，测试样品时，仪器提示结果是否保存或保存在什么文件夹时应选择附在刚才的文件夹下（标准曲线文件夹），如下图所示：



这样在测试报告后的 Data 栏中将出现 CSP 后缀名，用光标选择样品浓度和标准表，点击“Show report”，将显示结果报告。根据需要，选择是否打印或储存结果。

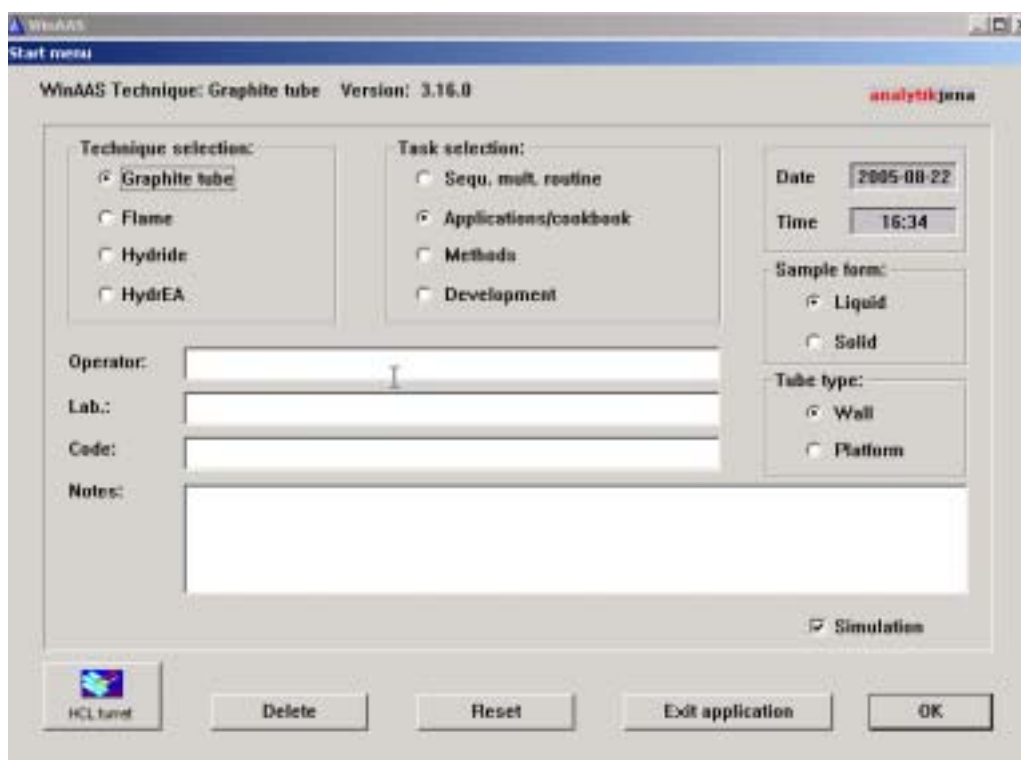
## 二、关机

- 1、“Flame”菜单中点击“Extinguish”熄灭火焰，或在主菜单中点击右手边的“Flame”熄灭火焰。或先关闭乙炔气瓶总阀，等火焰熄灭后，再退出 AAS 软件系统，此时乙炔气瓶分压表还有一定的压力是正常的。
- 2、退出 AAS 操作软件系统。
- 3、关闭 AAS ZEEnit700 主机电源，
- 4、关闭计算机电源。
- 5、关闭乙炔气瓶总阀，断开空气压缩机电源（将空气压缩机中的空气放掉）。
- 6、关闭电源总开关。

# 石墨炉模式

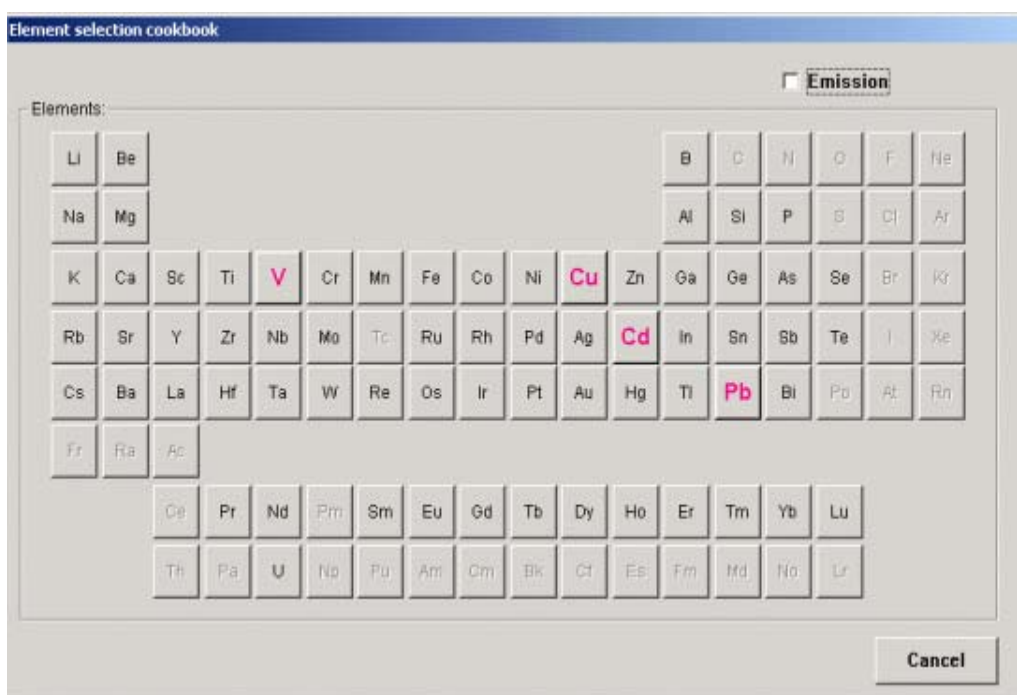
## 一、 开机

- 1、打开氩气气瓶总阀，调节气体减压阀使气体出口压力为 0.5Mpa 左右。
- 2、打开 KM5 水冷器电源开关（**注意：只有国产 KM5 才需打开其电源开关，进口水冷器无需打开电源**）。
- 3、打开 AAS ZEnit700 主机电源。
- 4、打开计算机电源。
- 5、双击 AAS 图标，进入应用程序的开始菜单界面：

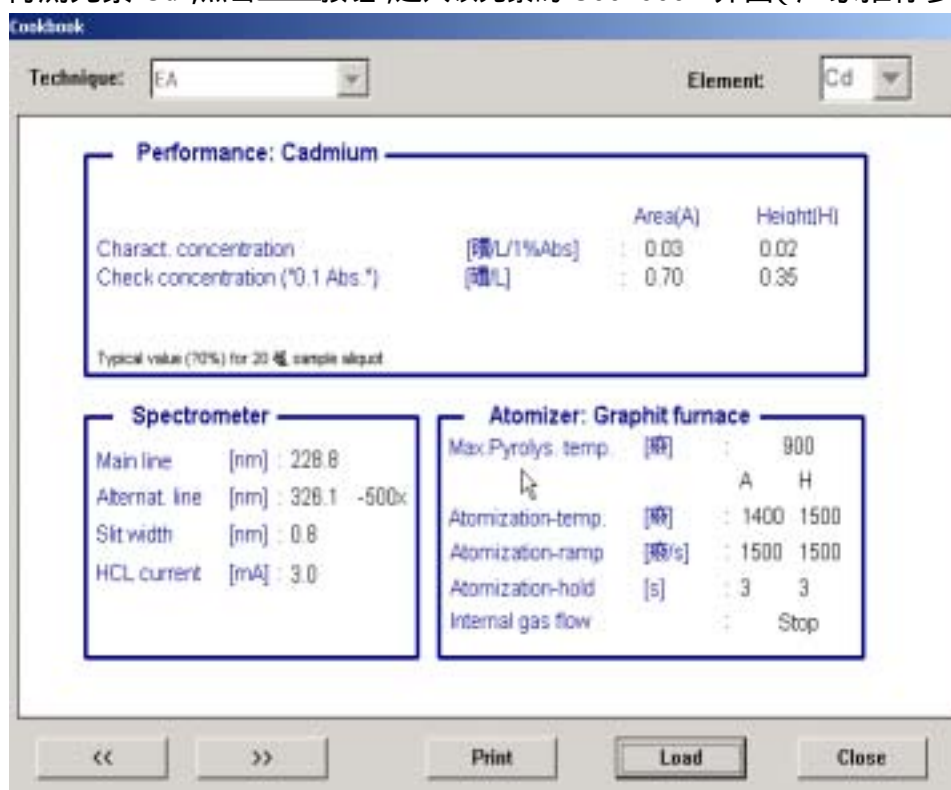


选择测量技术 (Technique selection) -----石墨炉 Graphite tube，选择分析任务 (Task selection) -----点击 Applications/Cookbook (厂家推荐参数) 或 Method(已存方法),选择石墨管类型----点击普通型 Wall 或平台型 Platform，最后点击 OK，进入下一个界面。

- 6、a.选择 Applications/Cookbook (厂家推荐参数)，点击 OK，出现以下界面：

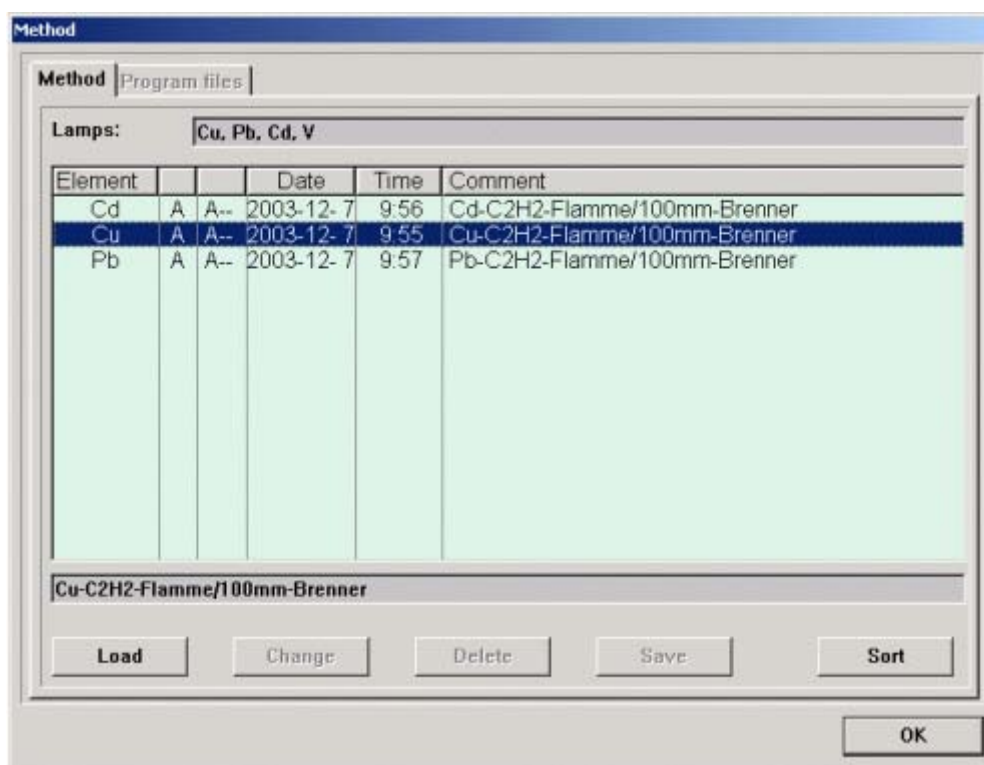


选择待测元素 Cd ,点击  按钮 ,进入该元素的 Cookbook 界面( 厂家推荐参数 )。



点击 Load 命令，进入初始化过程。

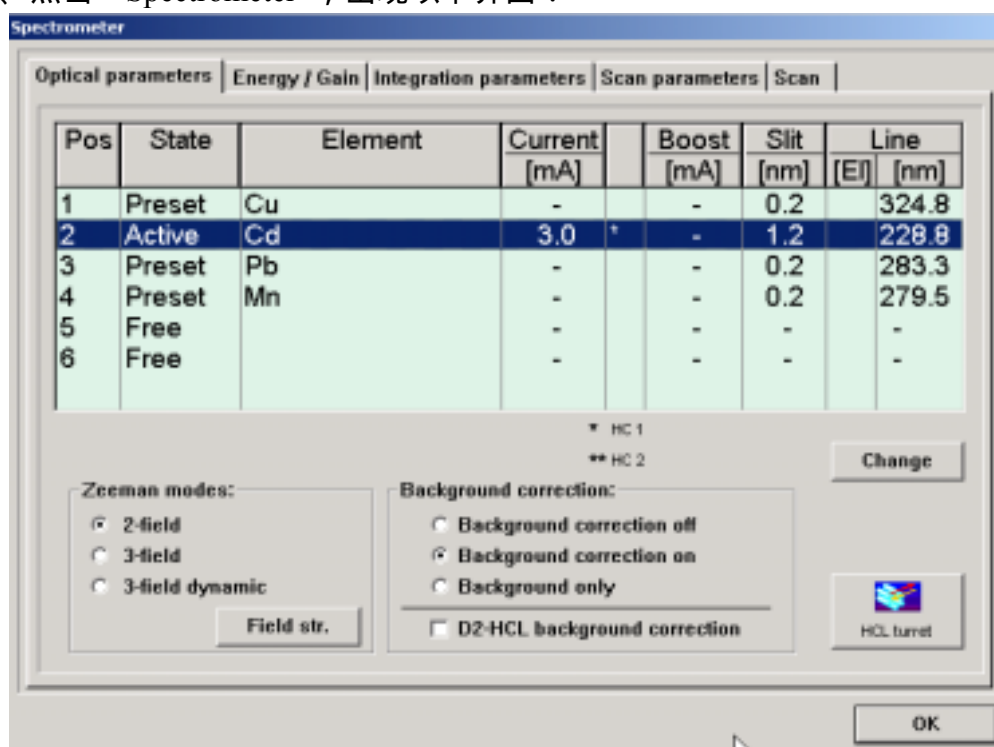
b.选择 Method(已存方法),点击 OK，进入下一个界面。



点蓝待测元素 Cd 所对应的方法，点击 Load 命令，进入初始化过程。

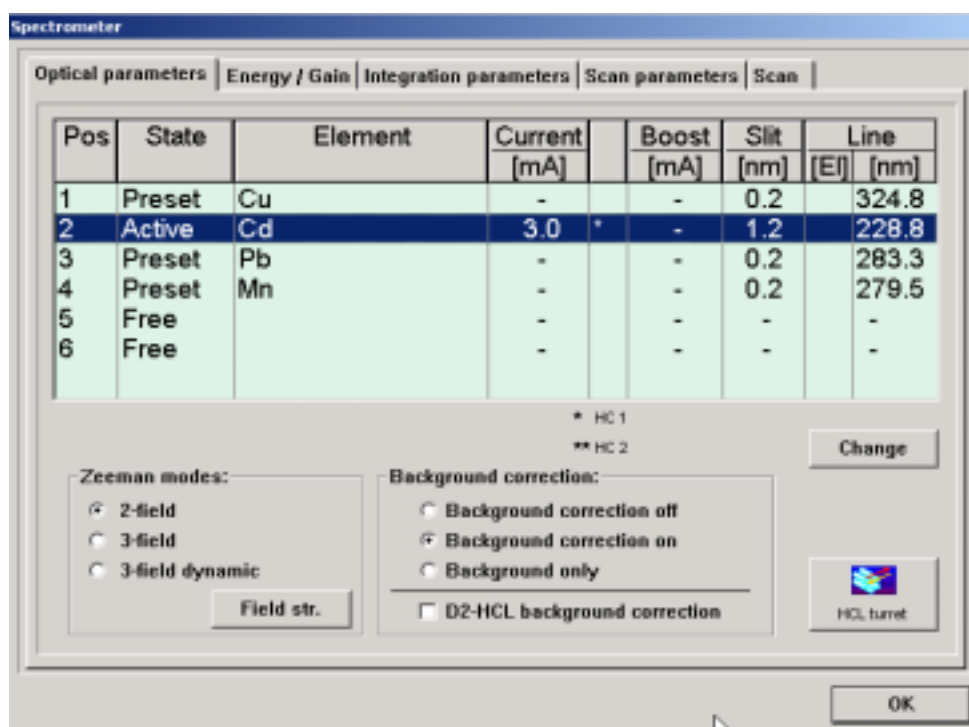
7、始化之后，首先调节自动进样器的注射位置（后面详述）。

8、点击“Spectrometer”，出现以下界面：

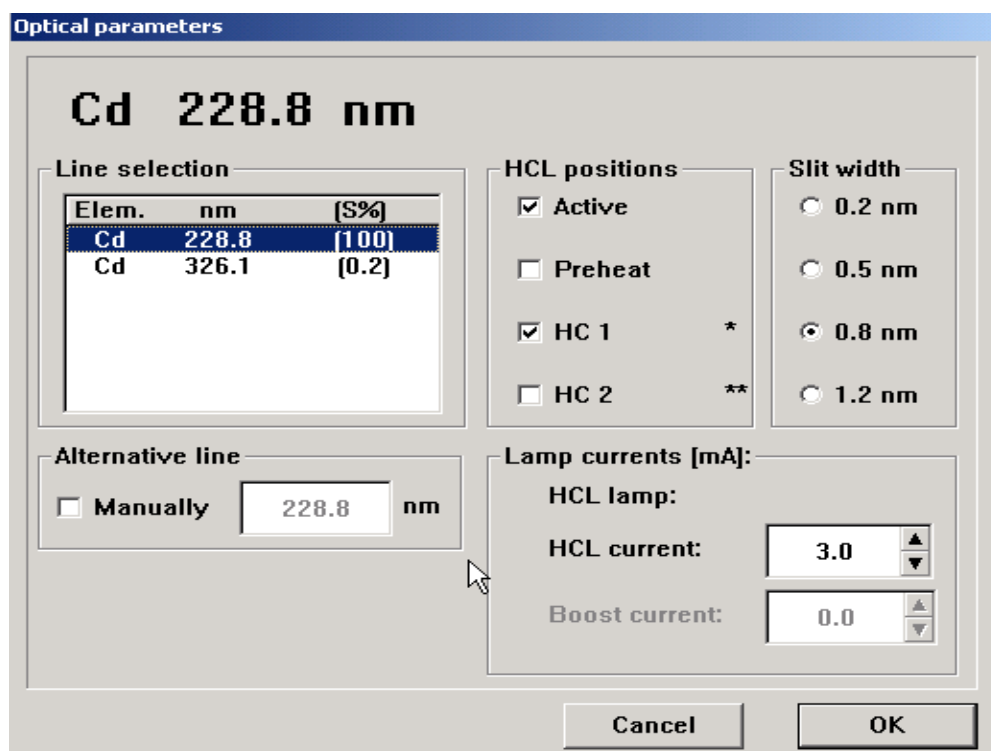


在“Spectrometer”界面内有五个标签：光学参数（Optical parameters），能量/增益（Energy/Gain），积分参数（Integration parameters），扫描参数（Scan parameters），扫描（Scan）。

(1)点击“Optical parameters”按钮，出现以下界面：



在这个界面里，点击 **Change** 按钮，进入光学参数界面：

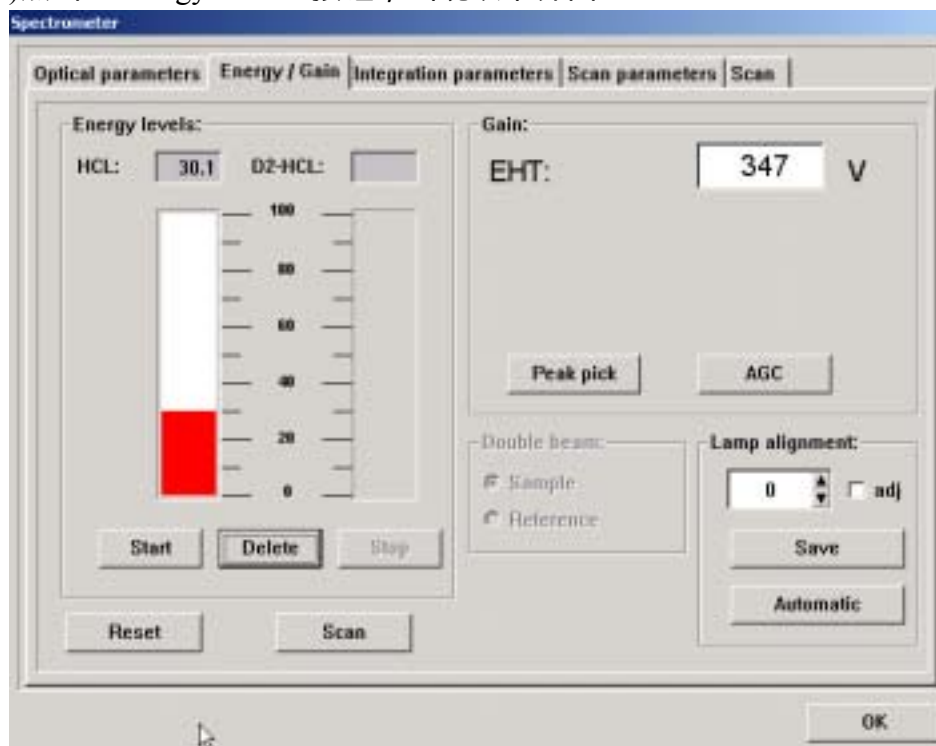


在光学参数界面内，可以选择或改变：元素分析线、灯的激活和预热状态、狭缝宽度、灯电流（调用 Cookbook 的参数时，如果使用的空心阴极灯是国产的，通常需要改变灯电流）。

另外你还可选择氩空心阴极灯扣背景，只需在

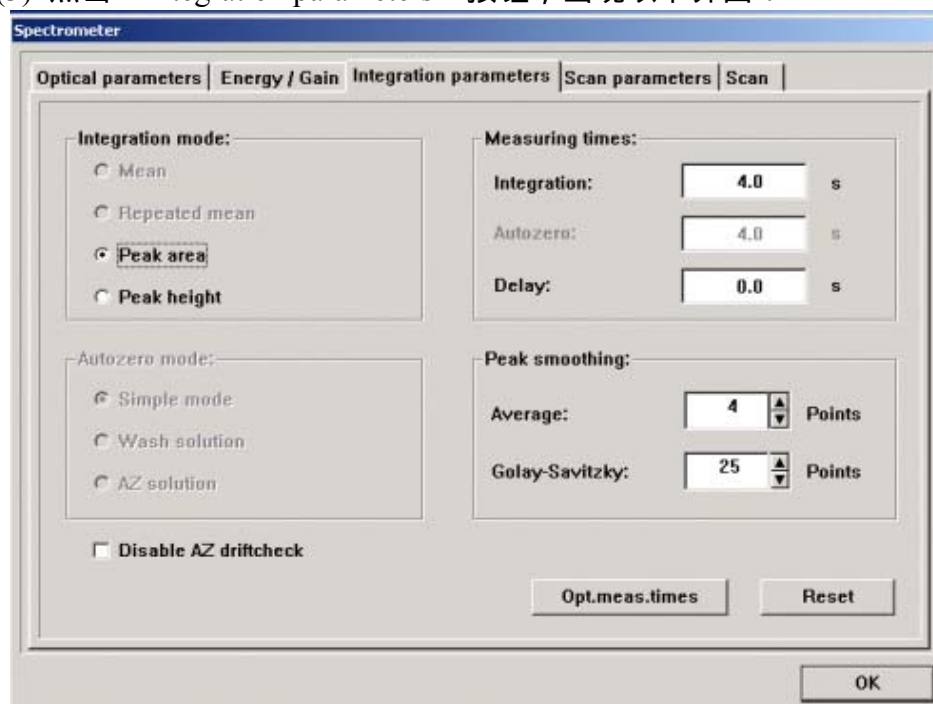
☒ **D2-HCL background correction** 前点击一下便可。

(2) 点击 “Energy/Gain ” 按钮，出现以下界面：



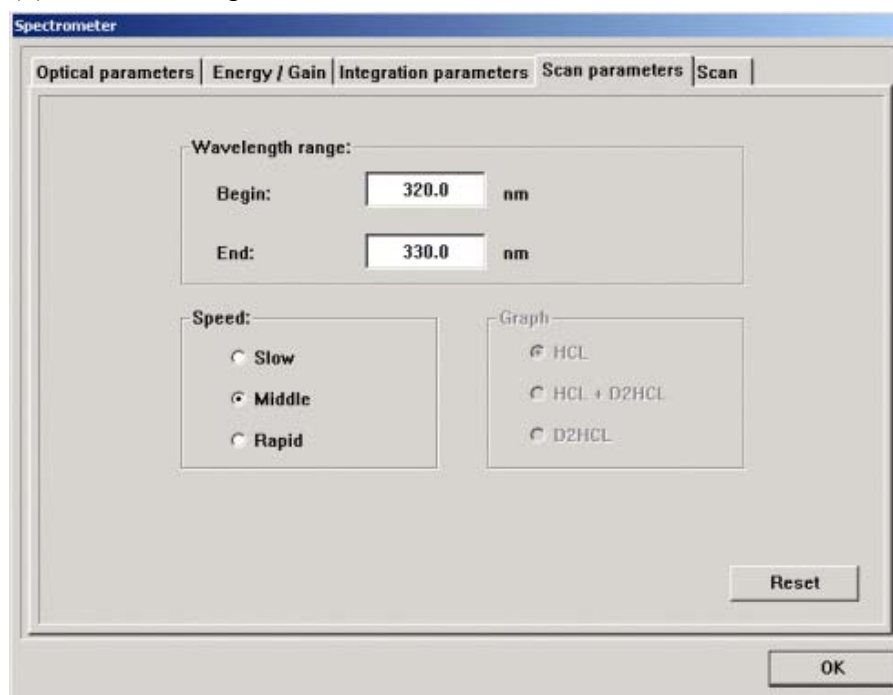
在 Lamp alignment 里，点击 Automatic 按钮，仪器开始自动调节待测元素空心阴极灯光源能量至最佳状态，并点击自动增益控制  按钮，使能量最佳化。

(3) 点击 “Integration parameters ” 按钮，出现以下界面：



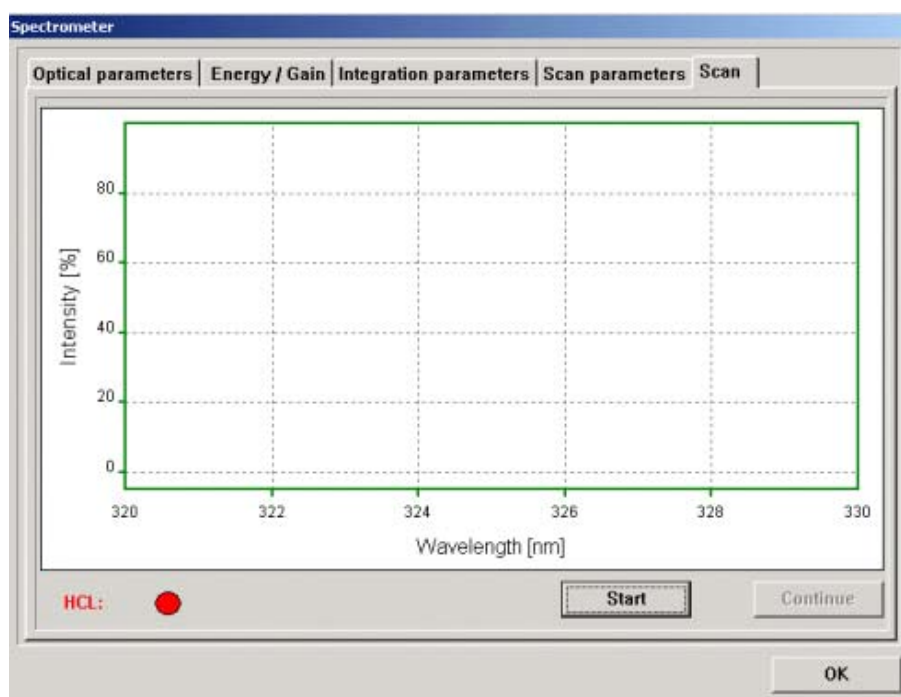
在积分参数界面，可根据以上数据设定。

(4) 点击 “ Scan parameters ” 按钮，出现以下界面：



在扫描参数界面里，输入扫描波长范围。输完之后，点击 SCAN 按钮进行波长扫描（此步可略）。

(5) 点击 “ Scan ” 按钮，出现以下界面：



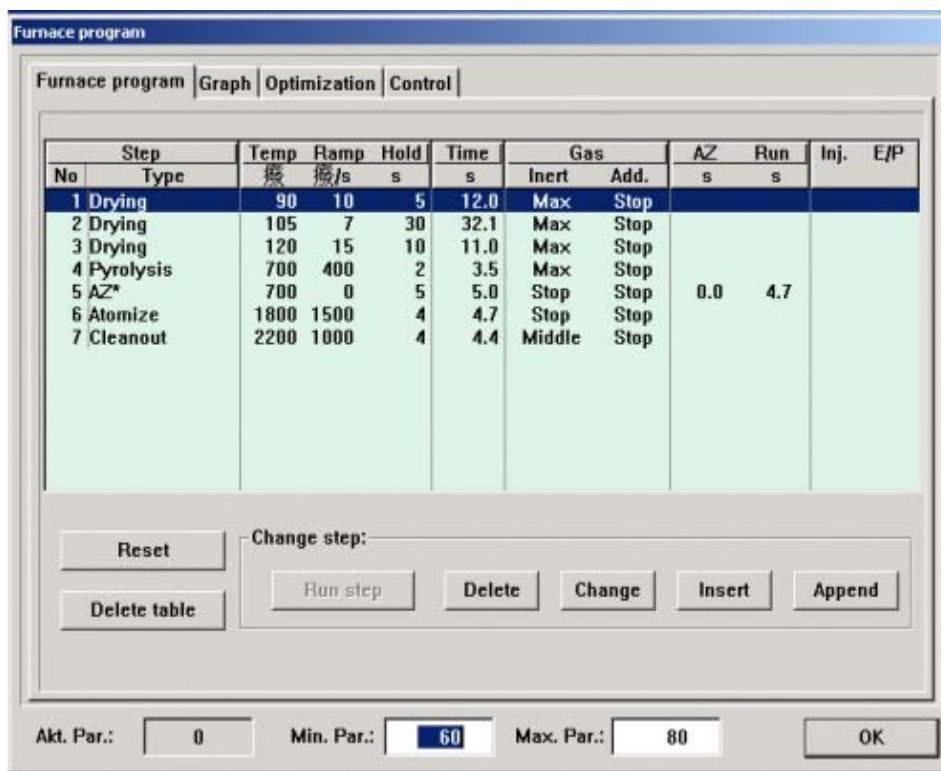
在扫描界面里，点击 START 按钮，开始波长扫描，扫描结束后，显示分析线波长（此步可略）。

最后点击 OK 键，退出 “ Spectrometer ” 界面。

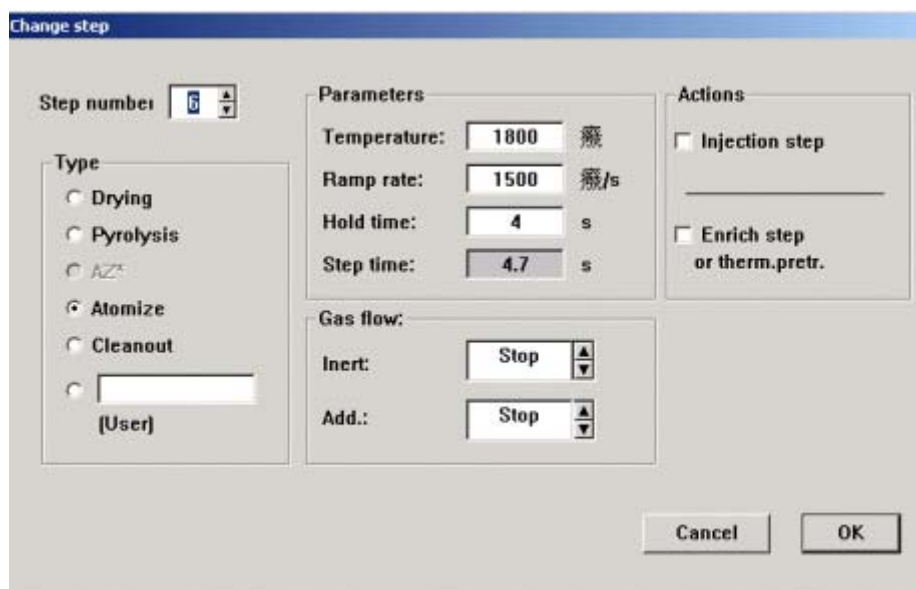
**注：最后两步一般只在质量鉴定时才使用，测试时狭缝宽度 slit 应选为 0.2nm。**

9、点击“Furnace”，进入石墨炉程序界面：

(1) 选择“Furnace program”，可以手动编辑石墨炉升温程序。



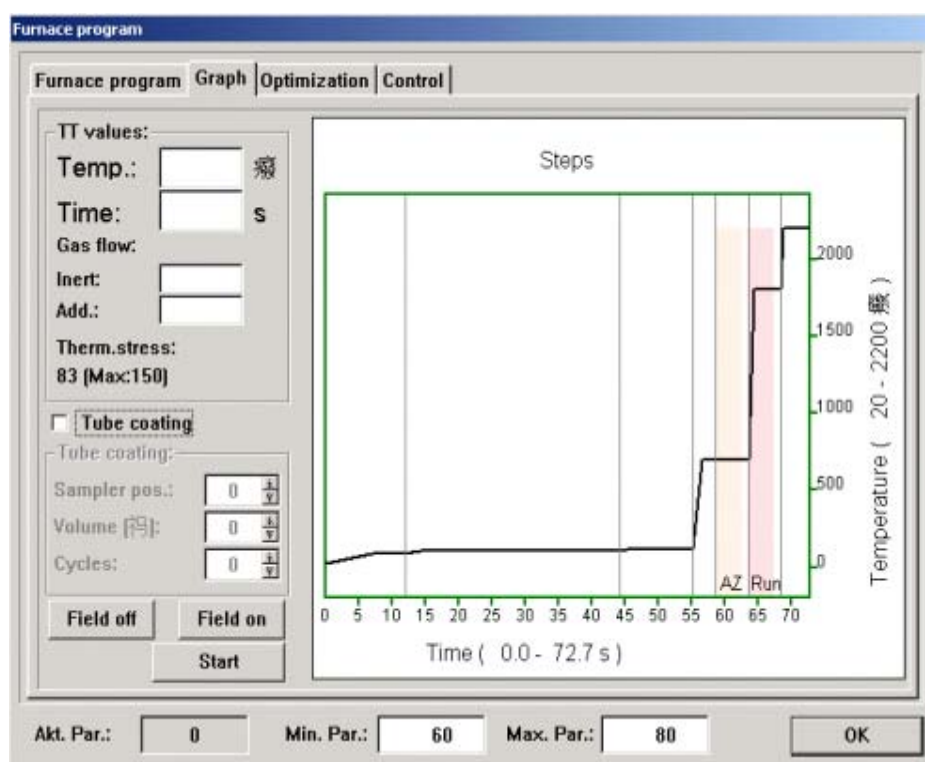
在石墨炉程序界面里，可以编辑石墨炉升温程序，包括：干燥(Drying)、灰化(Pyrolysis)、原子化(Atomize)、清洗(Cleanout)。双击升温程序的任一步骤，即可编辑。如双击原子化(Atomize)这一行，出现如下对话框：



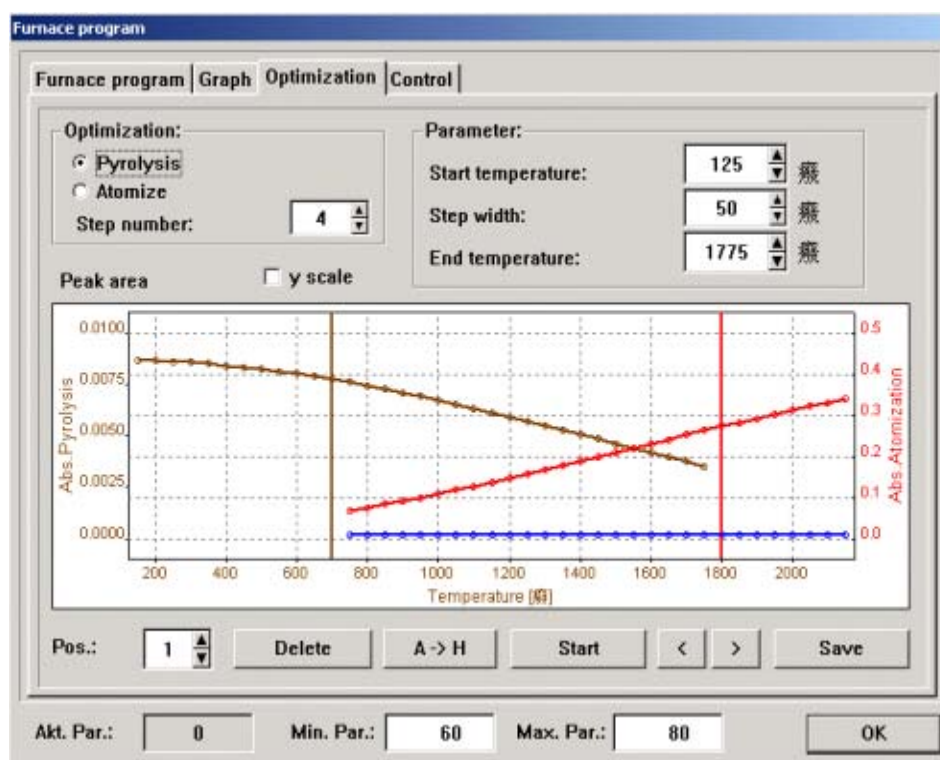
根据待测元素样品的实际情况，可以升高灰化温度、原子化温度、斜坡速率、保持时间等。

**注意：**当对样品没有经验时，请选用厂家推荐升温程序 (Cookbook)，同时应注意是否添加基体改进剂，选择适当的灰化温度及原子化温度。

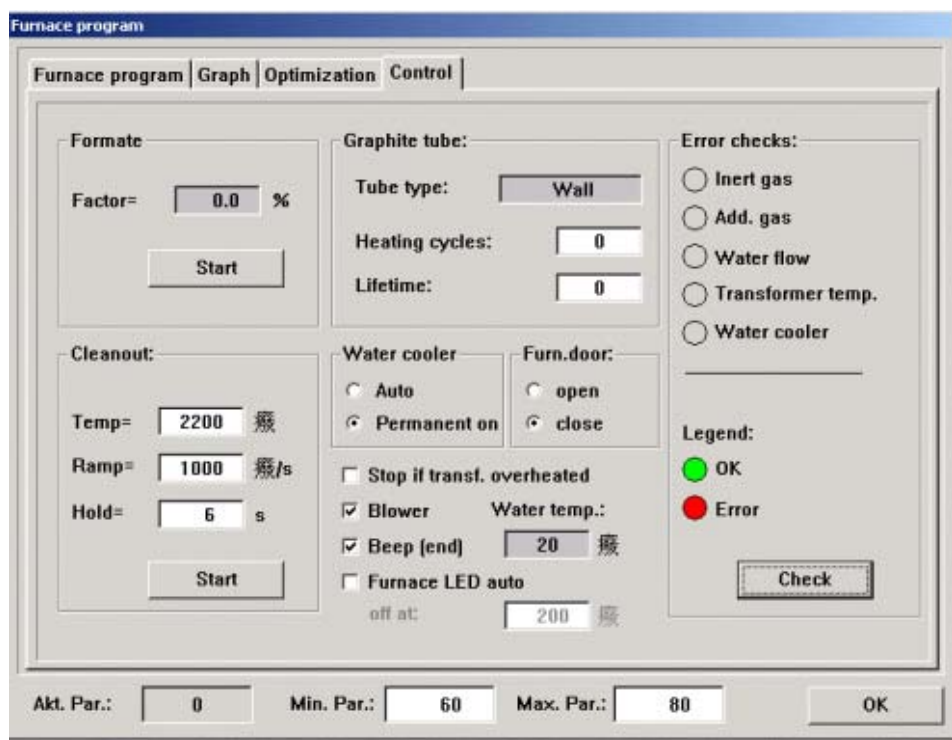
(2) 选择 “ Graph ”, 可以查看石墨炉升温程序的曲线轮廓：



(3) 选择 “ Optimization ”, 可以自动优化石墨炉升温程序。



(4) 选择 “ Control ”, 可以单独对石墨管进行控制。

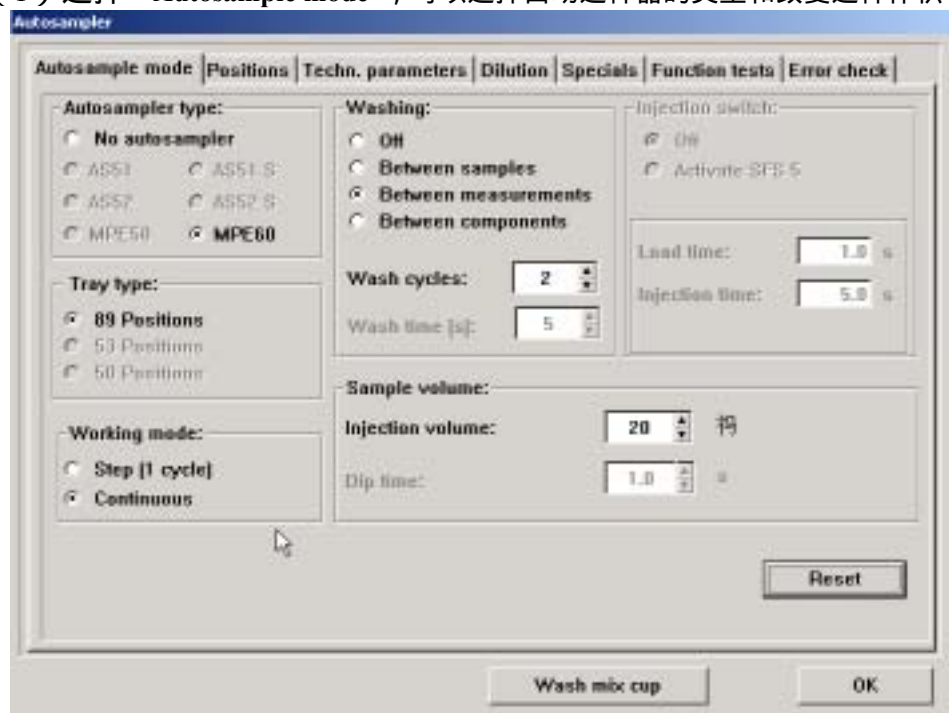


在该界面中，有格式化石墨管(Formate)、石墨管的加热次数(Heating cycles)和寿命评价(Lifetime)、对石墨管空烧控制(Cleanout)等。

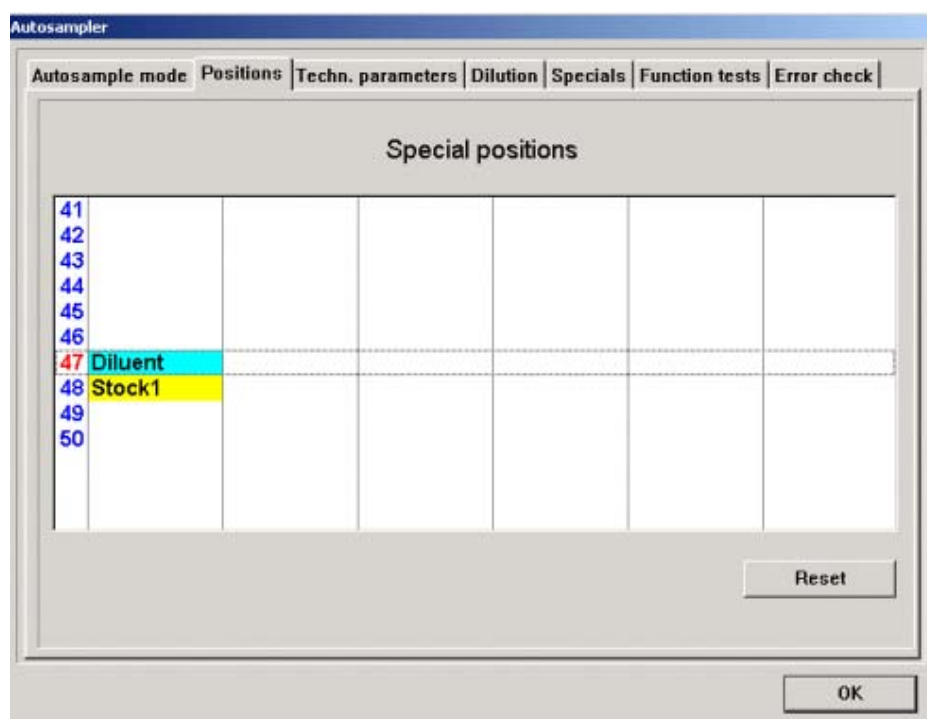
最后点击 OK 键，退出“Furnace”界面。

- 10、点击“Autosampler”，进入自动进样器的编辑界面。包括：自动进样器模式(Autosample mode)、样品瓶位置(Positions)、技术参数(Techn. Parameters)、稀释(Diluton)、特殊位置(Special)、功能测试(Function test)和误差检测(Error check)

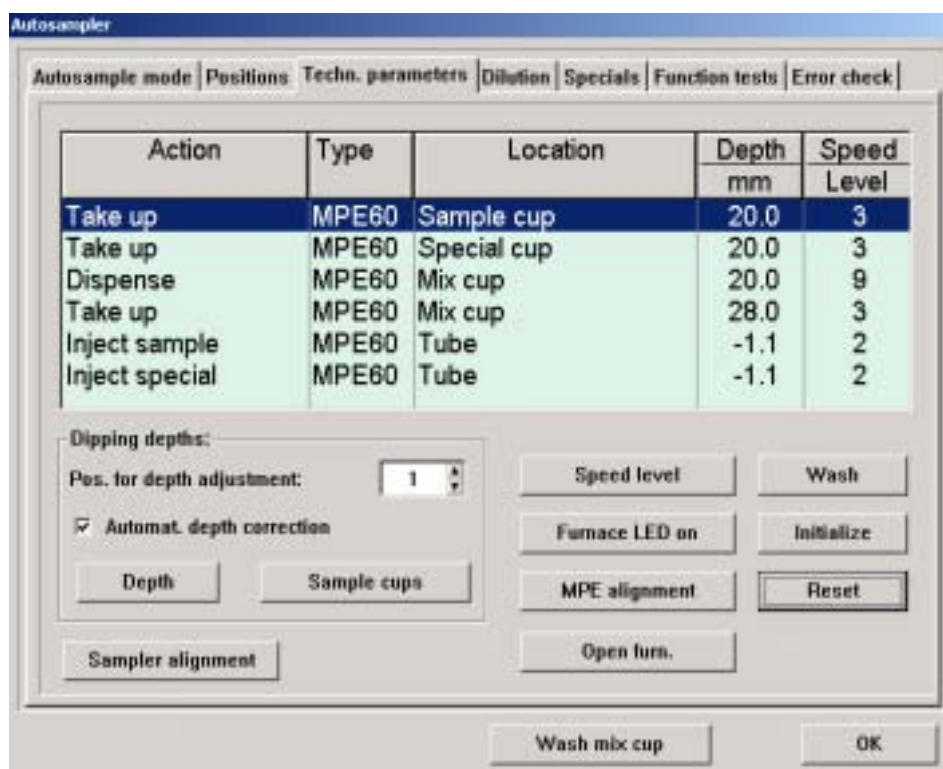
(1) 选择“Autosample mode”，可以选择自动进样器的类型和改变进样体积等。



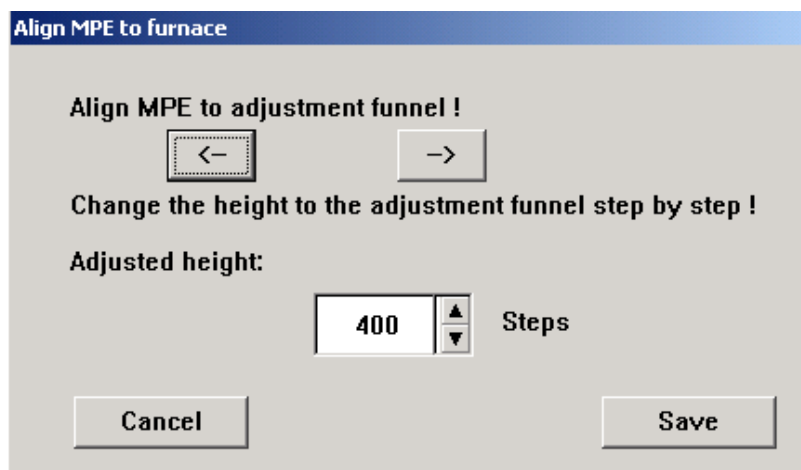
(2) 选择“Positions”，进入标样位置、稀释位置的编辑。



(3) 选择 “ Techn. Parameters ”，进入自动进样器的注射位置的调整。



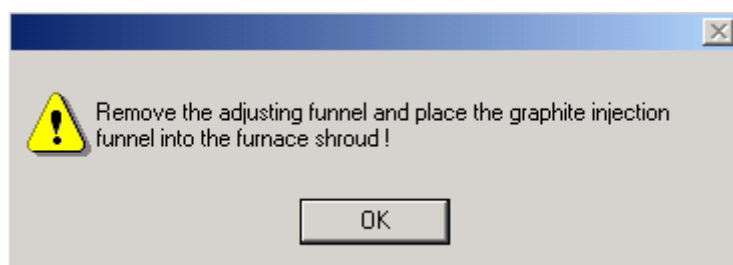
- A、 点击 **open furn.**，打开石墨炉，取出石墨管（已安装），取下左边石英窗，将 校正工具从左插入。
- B、点蓝 **Injection sample** 这一行，点击 **MPE alignment** 按钮，进入如下界面：



在 Adjusted height 中增大数字（如 420），调整样品进样管离校正工具上的小孔之间的距离，使样品进样管紧靠校正工具，点击  或  键，使样品进样管正好在校正工具上的小孔中心附近，然后再调整左右螺钉，使样品进样管正好在校正工具上的小孔中心，并锁紧左右螺钉。

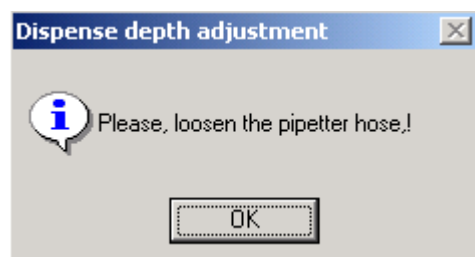
**注：调整之前应确保样品进样管超出保护导管不超过 8mm。**

C、点击 SAVE 按钮，进入下一界面：

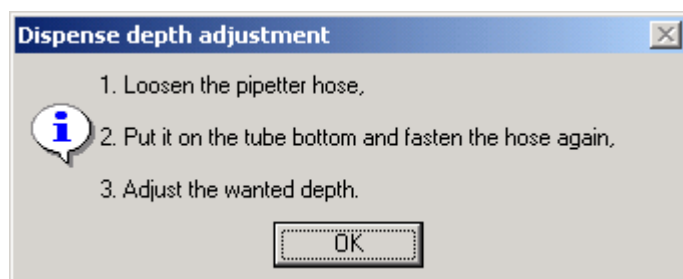


D、点击 OK，取出校正工具，插入石墨管，插入左边石英窗，点击 Close furn.，回到“Techn. Parameters”界面。

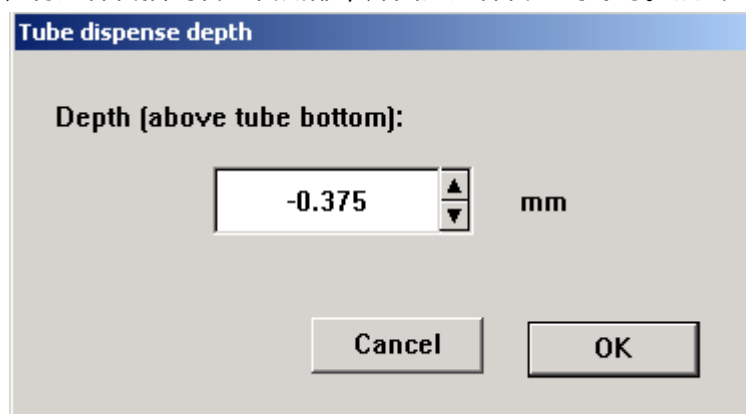
E、点击 ，点击进样管深度（Depth）按钮，进入以下界面：



F、松开进样管上的螺母，点击 OK，进入下一界面：



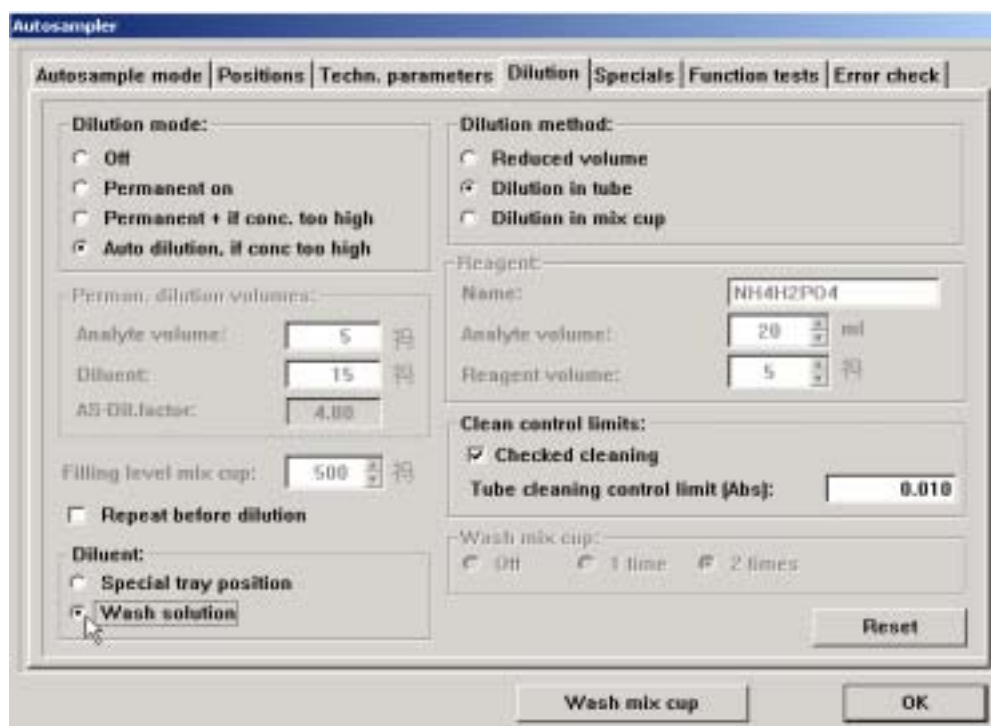
G、将进样管插到石墨管底部，并固定进样管上的螺母。点击 OK，进入下一界面：



用上箭头 ▲ 调整注射管与石墨管底部的距离。推荐为约-0.375 或-0.5mm。用牙医镜观察距离。

调整完毕后，点击 OK，结束调整过程。

（4）选择“**Diluton**”，可以设定稀释模式。当样品浓度太高时，选择是否自动稀释后，再测量。



注：选择在管内稀释 Dilution in Tube，最大可稀释 10 倍，选择在混合杯稀释 Dilution in mix cup 最大可稀释 625 倍，一般推荐选择在管内稀释 Dilution in Tube。

(5) 选择 “ Special ”, 可以设定基体改进剂是否添加。

The screenshot shows the 'Autosampler' window with the 'Specials' tab active. The 'Modifiers' section has three rows for #1, #2, and #3, each with a 'Name' field and a 'Vol. [μl]' spinner set to 10. The 'Enrichment in tube' section has a 'Mode' group with radio buttons for 'Off' (selected), 'Permanent [Samples only]', 'If conc. too low', and 'Permanent [incl. calib.]'. Below this is an 'Enrichment cycles' spinner set to 2 and a checkbox for 'Repeat before enrichment'. The 'Thermal pretreatment' section has an 'Activation' group with 'Off' (selected) and 'On'. The 'Warm up components' section has radio buttons for 'Sample' (selected), 'Modifier 1', 'Sample + Modifier 1', 'Modifier 1 + Sample', and 'Modifier 1 + Modifier 2'. A 'Warm up delay' spinner is set to 0. A 'Reset' button is at the bottom right of the main panel, and an 'OK' button is at the very bottom.

(6) 选择 “ Function test ”, “ Error check ”, 可以单独测试自动进样器的功能或误差检测 (此步可略)。

最后点击 OK, 退出自动进样器界面。

# 11、 标样测量 - 制作标准曲线。

- i. 点击 “ Calibration ”, 选择标准校正模式 ( Standard calibration ), 进入校正界面 :

The screenshot shows the 'Calibration' window with the 'Calibration mode' tab active. The 'Computation / calibration method' section has radio buttons for 'Absorbance mode (no calibration)', 'Standard calibration' (selected), 'Bracketing calibration', 'Method of addition', and 'Method of Additions calibration'. A 'Reset' button is located at the bottom right of the main panel, and an 'OK' button is at the very bottom.

在校正界面内，有校正模式(Standard mode)、条件设定(Conditions)、数学统计(Statistics)、标样浓度输入(Conc. Input)、标样运行表格(Table)、曲线参数(Curve parameters)。

ii. 点击“Conditions”，进入如下界面：

The screenshot shows the 'Calibration' window with the 'Conditions' tab active. The interface includes the following sections:

- Standards:**
  - Making standards:** Radio buttons for 'Using premixed standards', 'By different volumes', and 'By dilution' (selected).
  - No. of standards:** A numeric input field set to 5.
  - Sample meas. without add:** A numeric input field set to 3.
- Blank correction:**
  - Radio buttons for 'Absorbance corrected' (selected) and 'Concentration corrected'.
  - Checkboxes for 'Continue after curve calculation' and 'AZ [AZ/AM] between standards'.
- Getting values by:** Radio buttons for 'Measurement' (selected) and 'Manual input'.
- Mix conditions:**
  - Filling level mix cup:** A numeric input field set to 5 ml.
  - [sample part]:** A numeric input field set to 2.5 ml.
  - Sample volume:** A numeric input field set to 20 ml.
  - Max. add. volume:** A numeric input field set to 10 ml.
  - Total modifier vol.:** A numeric input field set to 0 ml.

Buttons for 'Reset', 'OK', and 'Cancel' are located at the bottom right.

在条件设定界面，可根据以上数据和选项设定。标准数量须根据实际情况而定。

iii. 点击“Statistics”，进入如下界面：

The screenshot shows the 'Calibration' window with the 'Statistics' tab active. The interface includes the following sections:

- Statistics:** Radio buttons for 'Off', 'Sigma statistics' (selected), and 'Median Statistics'.
- Measurement cycles:**
  - Runs/Std.:** A numeric input field set to 3.
  - Blind runs/Std.:** A numeric input field set to 1.
- Grubbs outlier test:** Radio buttons for 'Off' (selected), 'On (Flagging mean by "[I]")', and 'On [Without flagging]'.
- Statistical output:** Checkboxes for 'Standard deviation [SD]' and 'Relat. standard deviation [RSD]' (both selected).

Buttons for 'Reset', 'OK', and 'Cancel' are located at the bottom right.

在数学统计界面，可根据以上数据和选项设定。

iv. 点击“Conc. Input”，进入如下界面：

Calibration

Calibration mode | Conditions | Statistics | Conc. input | Table | Curve parameters | Recalibration

Stock standards

Kind of stock standards:

☒ Liquid  
☐ Solid

Concentrations:

|                                            | Conc.   |
|--------------------------------------------|---------|
| Stock solution 1:                          | 2.50000 |
| <input type="checkbox"/> Stock solution 2: | 1.0000  |
| <input type="checkbox"/> Stock solution 3: | 1.0000  |
| <input type="checkbox"/> Stock solution 4: | 1.0000  |

Concentration unit:

☐ mg/L  
☒  $\mu\text{g/L}$   
☐  $\mu\text{g/mL}$   
☐ ng/mL  
☐ ng/L

user defined

Conv.factor:

1000.00000

Absolute unit:

☐ mg  
☐  $\mu\text{g}$   
☒ ng  
☐ pg  
☐ fg

Reset

OK

在这个界面里，输入标样母液浓度如 2.50ppb，选择浓度单位即可,如 ( $\mu\text{g/L}$ )。

v. 点击“Table”，进入如下界面：

Calibration

Calibration mode | Conditions | Statistics | Conc. input | Table | Curve parameters | Recalibration

| Standards: |          |       |     | Vol. |    | Conc. | pKH |    |       |     |  |
|------------|----------|-------|-----|------|----|-------|-----|----|-------|-----|--|
| No         | Name     | State | Pos | 30   | D  | 20    | Abs | SD | RSD % | Abs |  |
| 1          | Cal-Zero | (-)   | 41  | 0    | 20 | 0.000 |     |    |       |     |  |
| 2          | Cal-Std1 | (1)   | 42  | 4    | 16 | 0.500 |     |    |       |     |  |
| 3          | Cal-Std2 | (1)   | 42  | 8    | 12 | 1.000 |     |    |       |     |  |
| 4          | Cal-Std3 | (1)   | 42  | 12   | 8  | 1.500 |     |    |       |     |  |
| 5          | Cal-Std4 | (1)   | 42  | 16   | 4  | 2.000 |     |    |       |     |  |
| 6          | Cal-Std5 | (1)   | 42  | 20   | 0  | 2.500 |     |    |       |     |  |
|            |          |       |     |      |    |       |     |    |       |     |  |
|            |          |       |     |      |    |       |     |    |       |     |  |
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|            |          |       |     |      | </ |       |     |    |       |     |  |

其中，Cal-Zero 为校正零点，即空白零点。

点击“Parameters”按钮或双击，进入标准系列浓度输入的对话框：

**Standard parameters**

Standard:

Sampler position:

**Making standards:**

Volume:  袞

from Stock std.:

☒ #1 (2.500 袞/L)    ☐ #3 (1.000 袞/L)  
☐ #2 (1.000 袞/L)    ☐ #4 (1.000 袞/L)

Concentration:  袞/L

Absorbance:

P<sub>KH</sub>                      P<sub>KA</sub>

依次输入吸取标准体积如 16ul，直到输完为止。

vi. 点击 “**Curve parameters**”，进入如下界面：

**Calibration**

Calibration mode | Conditions | Statistics | Conc. input | Table | **Curve parameters** | Recalibration

**Curve calculation:**

Curve form:

☐ Linear  
☐ Nonlinear  
☒ Automatically

Intercept:

☐ Zero  
☒ Calculate

**Weights of calibration points:**

☒ No weights  
☐ 1 / Conc  
☐ 1 / SD  
☐ 1 / (SD \* Conc)

**Curve examination:**

☒ No outlier test  
☐ Flag and eliminate outlier  
☐ Repeat measurement of the outlier

在这个界面面，可根据以上选项设定。

vii. 重新点击 “**Table**”，进入如下界面：

Calibration

Calibration mode | Conditions | Statistics | Conc. input | Table | Curve parameters | Recalibration

| Standards: |          |       |     | Vol. |    | Conc. | Pkh |    |       |     |
|------------|----------|-------|-----|------|----|-------|-----|----|-------|-----|
| No         | Name     | State | Pos | μL   | D  | μg/L  | Abs | SD | RSD % | Abs |
| 1          | Cal-Zero | (-)   | 41  | 0    | 20 | 0.000 |     |    |       |     |
| 2          | Cal-Std1 | (1)   | 42  | 4    | 16 | 0.500 |     |    |       |     |
| 3          | Cal-Std2 | (1)   | 42  | 8    | 12 | 1.000 |     |    |       |     |
| 4          | Cal-Std3 | (1)   | 42  | 12   | 8  | 1.500 |     |    |       |     |
| 5          | Cal-Std4 | (1)   | 42  | 16   | 4  | 2.000 |     |    |       |     |
| 6          | Cal-Std5 | (1)   | 42  | 20   | 0  | 2.500 |     |    |       |     |

Clear Table | Set recal | Check | Run sample

Delete line | Parameters | Fit curve | → Blindw.

OK

**Cd**  
228.8 /Max

Open fum.  
Cleanout  
Wash  
Formate  
Continue  
Start/Abs

在这个界面里，点击“Start/Abs”，开始标样的自动测量。  
接下来，会提示测量的信息：

Furnace report

Step: 1 (Drying)

Temp.: 90 °C

Time: 10 s

Ramp rate: 10 °C/s

Gas: Max

干燥过程

Furnace report

Step: 4 (Pyrolysis)

Temp.: 300 °C

Time: 56 s

Ramp rate: 300 °C/s

Gas: Max

灰化过程

**Furnace report**

Step: 5 [AZ\*]

Temp.: 300 癩

Time: 60 s

Ramp rate: 300 癩/s

Gas: Stop

原子化过程

**Cleanout**

Step: 1 [CLEAN]

Temp.: 2 000 癩

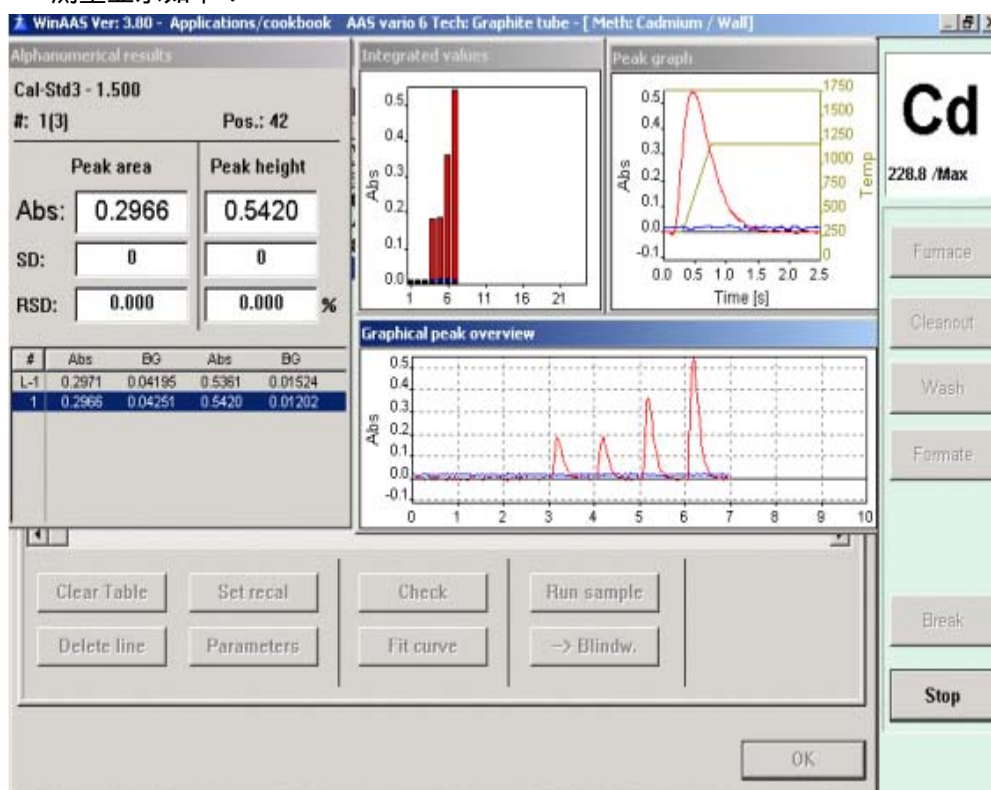
Time: 2 s

Ramp rate: 1000 癩/s

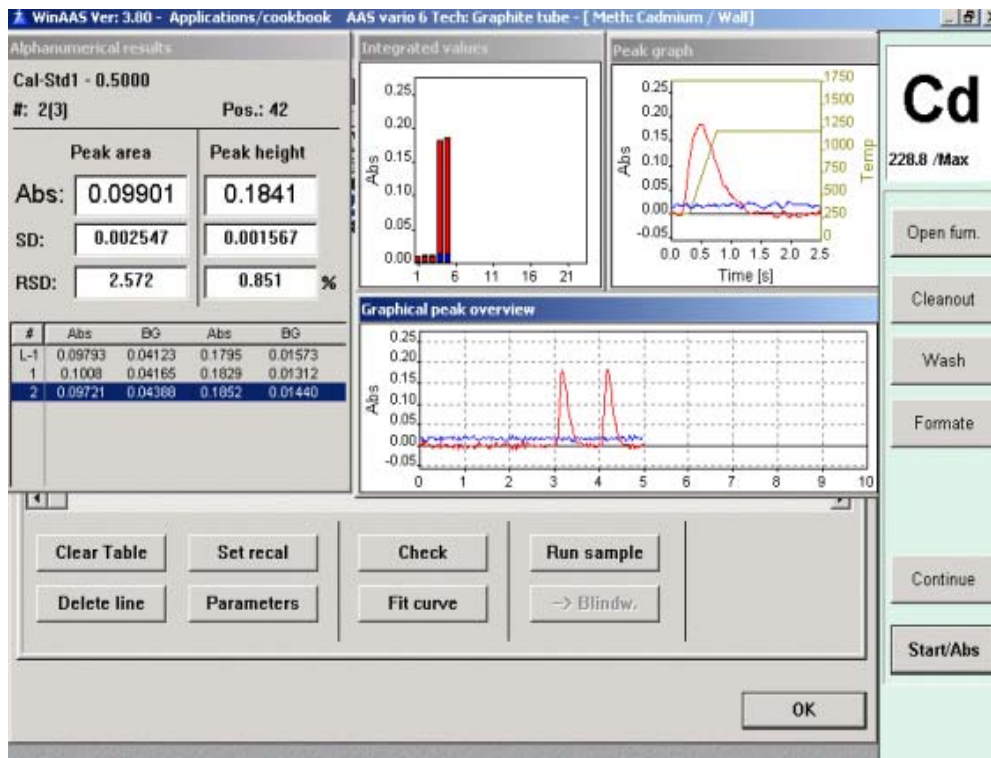
Gas: Max

清洗过程。由于清洗过程较短，一般看不到该界面。

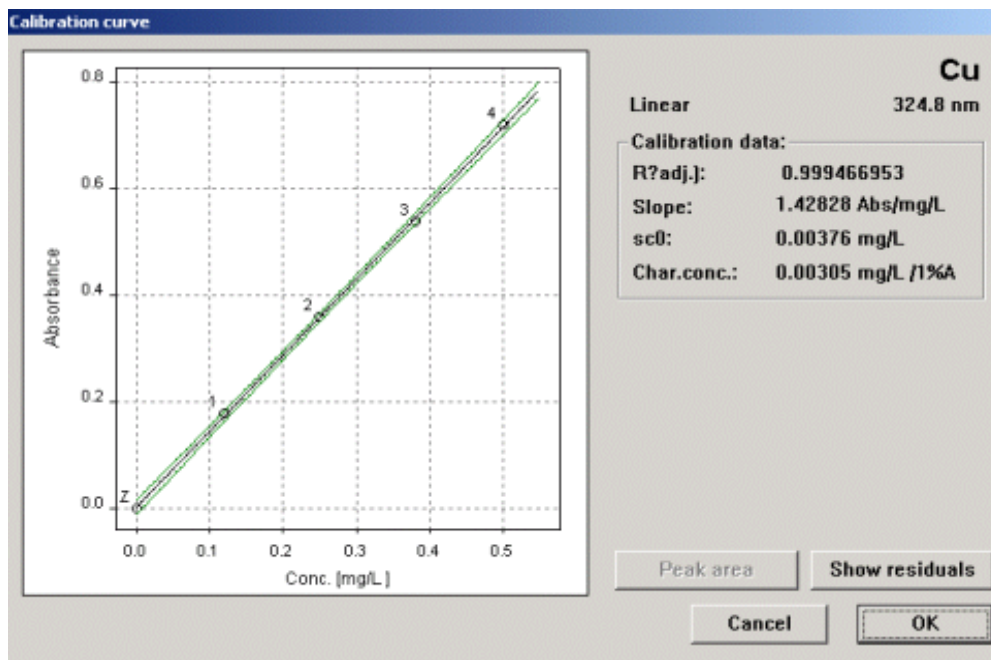
测量显示如下：



测量完毕提示如下：



当所有的标样测量完毕时，点击“Fit curve”查看标准曲线的相关系数：



如果  $R^2 \geq 0.995$ ，标准曲线良好，可以进入待测样品测量。否则，需删除某个点或另配标样，点击“Run sample”，重新单测量不准确的标样。

最后点击 OK 键，退出“Calibration”界面。

## 12、 样品测量。

- 点击“Samples”，进入样品测量界面：



WinAAS Ver: 3.80 - Applications/cookbook AAS vario 6 Tech: Graphite tube - [Meth: Cadmium / Wall]

Samples

Sample table | Conc. output | ID samples | Statistics | Mult.Param.

Concentration output [Conc2] for:

☒ Liquid original sample

☐ Solid original sample

Output style:

Absorbances:

☐ 3 signif. digits

☒ 4 signif. digits

☐ 5 signif. digits

Concentrations:

☐ 3 signif. digits

☒ 4 signif. digits

☐ 5 signif. digits

Concentrations:

☐ mg/L

☒ µg/L

☐ µg/mL

☐ ng/mL

☐ ng/L

☐ user defined

Conv.factor: 1.000000000

Reset

OK

在这个界面，可根据以上选项设定。

iii. 点击“ID sample”，点击“Sample table → ID”，出现如下界面：

Samples

Sample table | Conc. output | ID samples | Statistics | Mult.Param.

Sample ID-Fixed part:

Batch:

Customer order:

Sample ID / Fixed part:

Solution of solid samples:

Sol. volume: 100 ml

Nominal weight: 1.00 g

| No | Pos | Name  | Pre-DF | Vol.[mL] | Wt[g] | Blank corr. |
|----|-----|-------|--------|----------|-------|-------------|
| 1  | 1   | WATER | 1.000  |          |       | Off         |
| 2  | 2   | Cd    | 1.000  |          |       | Off         |
| 3  | 2   |       | 1.000  |          |       | Off         |
| 4  | 3   |       | 1.000  |          |       | Off         |
| 5  | 4   |       | 1.000  |          |       | Off         |
| 6  | 5   |       | 1.000  |          |       | Off         |
| 7  | 6   |       | 1.000  |          |       | Off         |
| 8  | 7   |       | 1.000  |          |       | Off         |
| 9  | 8   |       | 1.000  |          |       | Off         |
| 10 | 9   |       | 1.000  |          |       | Off         |
| 11 | 10  |       | 1.000  |          |       | Off         |

Sample table → ID | Change line | Insert line | Save | → Sample table

Delete table | Delete | Append line | Load | Synopsis

OK

iv. 双击其中一行，出现如下界面：

**Sample parameters**

Table row number:

ID/Wt data:

Autosampler cup position:

Sample name:

Pre-dilution factor:

Sol. volume:  ml

Weight:

Relative humidity:  %

☐ Blank correction

在这个界面中，输入样品名称，如 WATER。

v. 点击“Statistics”，进入如下界面：

**Samples**

Sample table | Conc. output | ID samples | **Statistics** | Mult.Param.

Statistics:

☐ Off

☒ Sigma statistics

☐ Median Statistics

Cycles:

Measurement cycles:

Runs/QC sample:

Blind cycles:

Statistical output:

☒ Standard deviation (SD)

☒ Relat. standard deviation (RSD)

☐ Relative homogeneous factor (RHF)

Grubbs outlier test:

☒ Off

☐ On [Flagging mean by "[I]"]

☐ On [Without flagging]

Confidence interval calculation:

☒ Off

☐ Absolute

☐ Relative

Confidence levels:

☐ 68.3 % [1 Sigma]

☐ 90 %

☐ 95 %

☒ 95.4 % [2 Sigma]

☐ 98 %

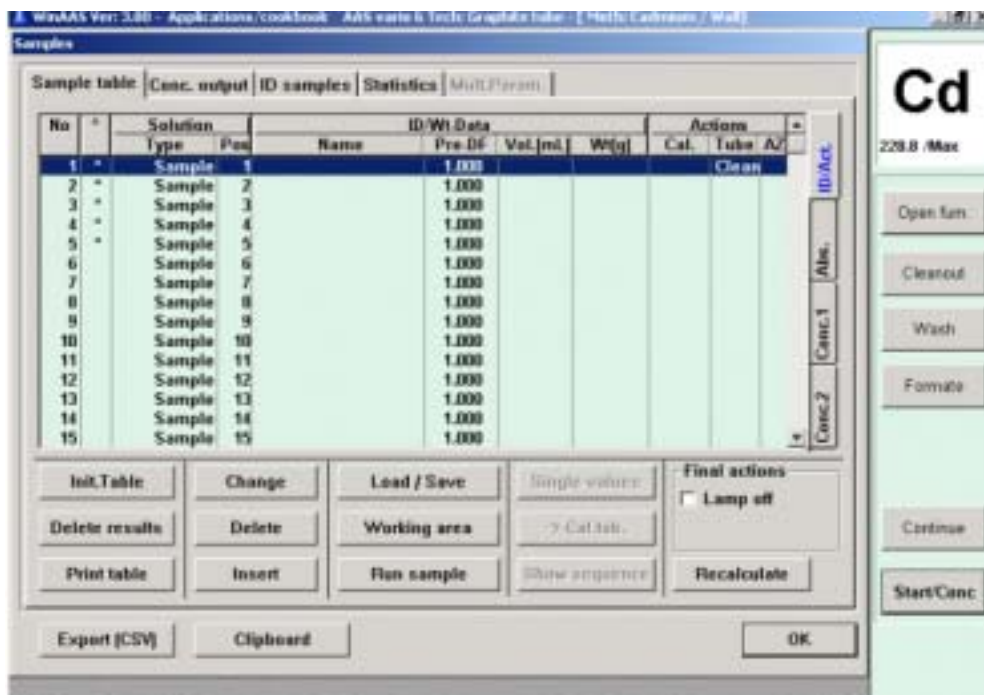
☐ 99 %

☐ 99.7 % [3 Sigma]

☐ 99.9 %

在这个界面，可根据以上数据和选项设定。

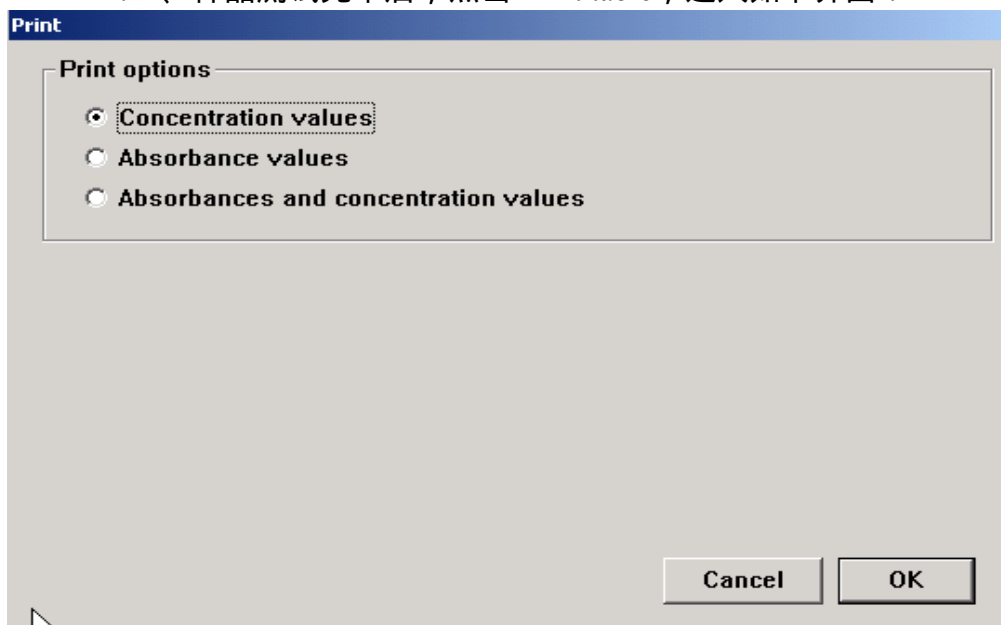
vi. 重新点击“Samples”，进入样品测量界面：



点击“Start/Conc”，连续测量或“Run sample”单个测量光标所在的样品。测量界面类似标样测量。

### 13、 测试结果打印。

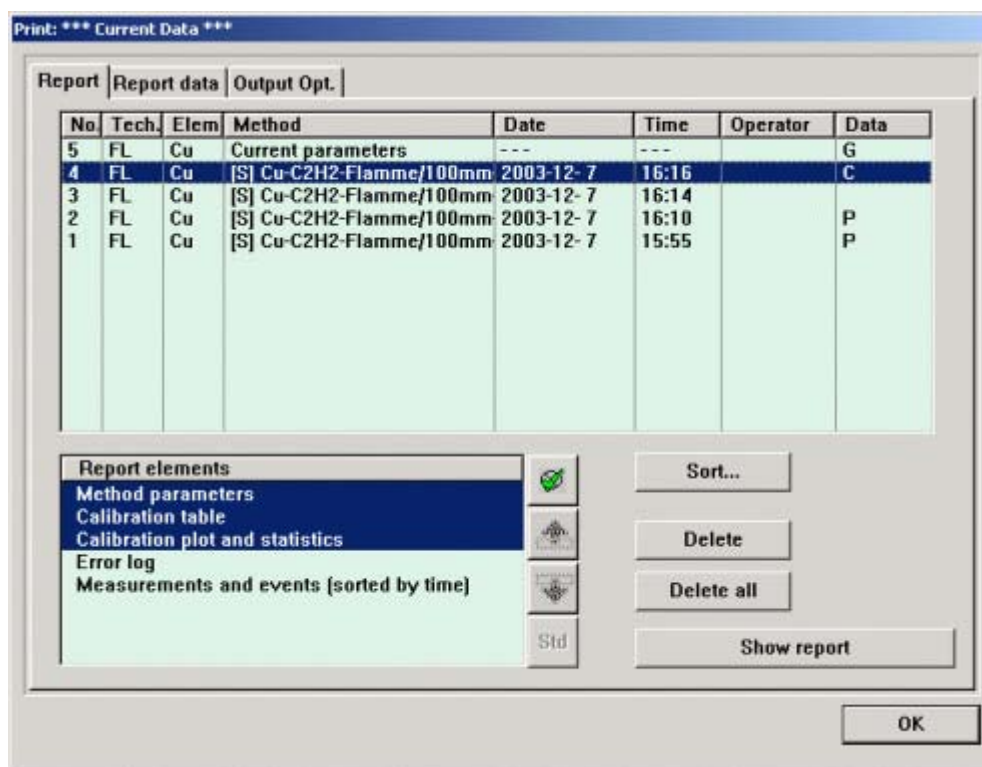
1. 、样品测试完毕后，点击 Print table，进入如下界面：



选择打印吸光值或/和浓度值报表。

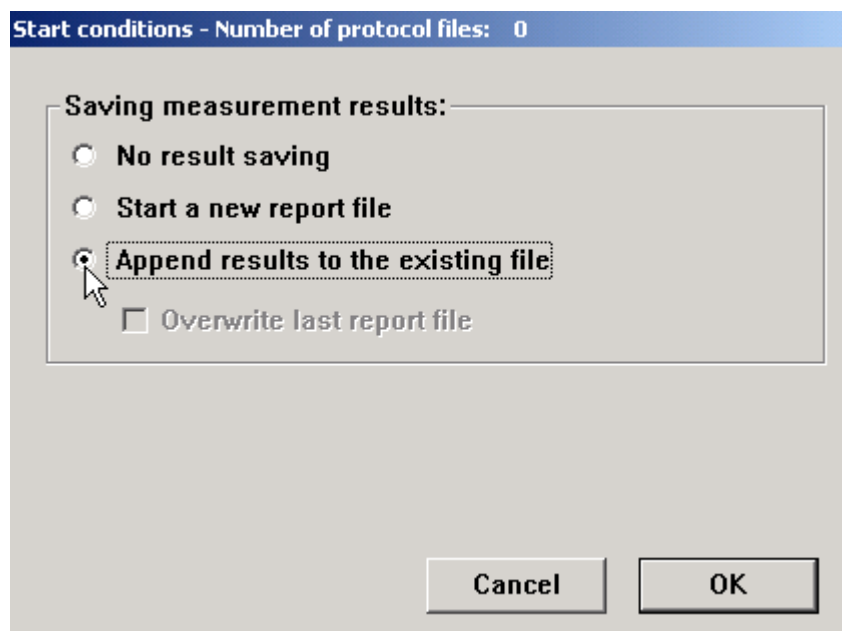
测试结果转存：点击 **CSV file**，可将结果转存到任何路径下保存，并可通过 Excel 文档对测试结果进行编辑。

## 2. 点击 “ Print ” 命令 ,



点击 “ Show report ”，将显示结果报告。根据需要，选择是否打印或储存结果。

注：若需将测试结果与标准曲线打印在同一张纸上，做完标准曲线后，测试样品时，仪器提示结果是否保存或保存在什么文件夹时应选择附在刚才的文件夹下（标准曲线文件夹），如下图所示：



这样在测试报告后的 Data 栏中将出现 CSP 后缀名，用光标选择样品浓度和标准表，点击 “ Show report ”，将显示结果报告。根据需要，选择是否打印或储存结果。

## 二、关机

- 1、 点击按钮，退出 AAS 操作软件。
- 2、 关闭计算机电源。
- 3、 关闭 AAS ZEE nit700 主机电源。
- 4、 关闭氩气气瓶总阀。
- 5、 关闭水冷器电源（**注意：只有国产 KM5 才需关闭其电源开关，进口水冷器无需关闭其电源开关**）。
- 6、 关闭电源总开关。

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