TURNING ON THE INSTRUMENT:

- Turn on the computer and the **Varian AA240FS**.
- Ensure exhaust is working and that acetylene pressure is above 700 kPa (~100 psi).
- Open the acetylene and air gas valve to the following settings:

Acetylene	11 psi
Air	45 psi

- Open the **SpectrAA** software and load a worksheet.

SETTING UP THE EXPERIMENT:

- Experiment set up is carried out by generating a worksheet to match the experimental conditions and lamp requirements. A worksheet can be generated by modifying an existing one **New from**, or by opening a **New** worksheet.
- Input the **Name** of the experiment, the **Analyst**'s name, any **Comments** and the **Number of samples** (default is 50). Save in Data\QuaterYear\Class folder.
- The worksheet will now allow to modify method and sequence parameters. In the **Develop** tab, click on **Add Method** and select the method of interest. This can be done from the software's **Cookbook**, which contains default parameters or from a **Method Library** previously developed. Select **Flame** under method type.
- For multi-element analysis continue adding methods to match the analyte. The instrument will analyze each sample for all elements of interest before proceeding to the next sample.

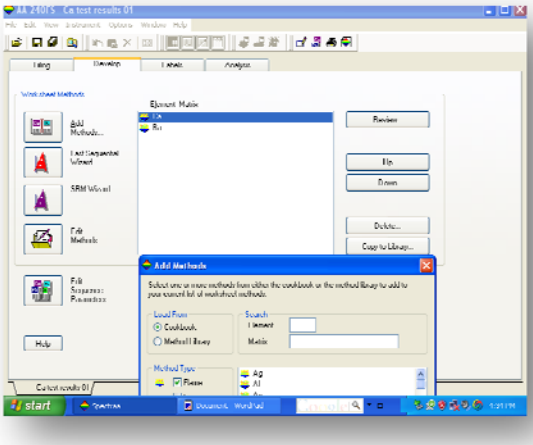

Figure 2. Method Development.

- Make sure lamp current and position match the method settings. It is possible enter this values in **Edit Methods**; setting can also be inspected by clicking on **Review.**
- Under the **Edit Method** menu several tabs are available:

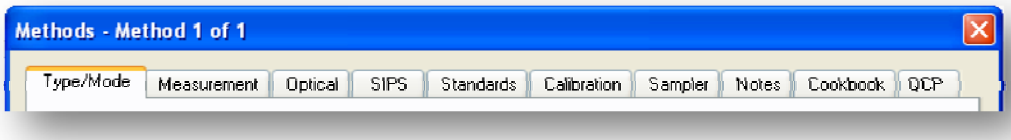

Figure 3. Method Editing.

- Under **Type/Mode** tab, the following settings can be altered:

Heading	**Options**	**Sample Settings**
Method	Type Element	Flame Ca, Ba etc…
Sampling Mode	Manual Autonormal	Manual
Instrument Mode	Absorbance Emission	Absorbance
Flame type & Gas flows	Flame Type Air Flow Acetylene Flow	Air/Acetylene 13.50 2.00
Online Diluter Type	Use SIPS Sampler Dilutor	Use SIPS

- Under the **Measurement** tab, the following settings can be altered:

Heading	**Options**	**Sample Settings**
Measurement Mode	PROMT Integration Integrate Repeat	PROMT
Calibration Mode	Concentration Standard Additions Scale Expansion	Concentration
Times	Measurement Read Delay	10.0 10
Replicates	Standard Sample	3 3
Precision (%)	Standard Sample	1.0 1.0

- Under the **Optical** tab, the following settings can be altered:

Heading	Options	Sample Settings
Lamp Position	Match position with location of lamp on carousel	Ca #4
Lamp Current (mA)	Match current with lamp requirement	10.0 mA
Wavelenght	Match wavelenght with lamp requirement	422.7 nm
Slit	Match slit with lamp requirement	0.5 nm
Backgorund	Enter desired correction	BC Off

- Under the **SIPS** tab, the following settings can be altered:

Heading	Options	Sample Settings
Nebulizer Uptake Rate	Units in mL/min	5.0 mL/min
Right Pump	None	None
Standard Additions	Linear Reading Range Calculate LRR	Unselect
Calibration Mode	Auto Set Std Concentrations Manually Entered Standards Use Premixed Standards	Auto Set Std Concentrations
Dual Pump Calibration	With ReCalibration With Reslope	Unselect

- Under the **Standards** tab, a list of the standards automatically generated will appear. In this tab it is possible to set the **Upper and Lower Valid Concentration** values, the units and decimal places as well as enter the **Bulk Standard Concentration** and **the Concentration Count**, which determines the number of samples run for calibration.
- The **Calibration** tab allows to set the **Calibration Algorithm** used to fit the calibration results, the **Recalibration Rate** (number of samples after which the instrument will automatically recalibrate), the **Reslope Rate** (number of samples after which the instrument will Reslope the data) as well setting up slope test parameters.
- The **Cookbook** tab provides information about alternative wavelengths and known interferences from other elements.
- For routine analyses the remaining tabs can remain unaltered with default settings.
- Exit the Method Edit menu and click on the **Labels** tab. Here the sequence can be edited by naming each sample, adding or deleting rows as well as repeating samples. Nominal weigh and volumes can be altered to indicate sample composition, but the settings do not affect the analysis. Sampler Racks and Carousel are not currently available for this instrument.

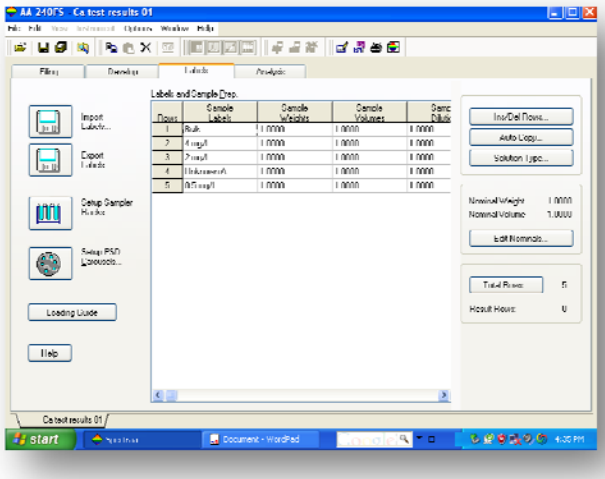

Figure 4. Labels Tab.

DATA COLLECTION:

- To align the burner: use the Varian supplied burner cleaning and alignment card to locate the light path. Rotate the burner by squeezing the prongs of the rotation handle, until the slot is parallel to the light path.
- Place the card halfway along the burner slot. Position the card with the vertical line perpendicular to the slot, and adjust the burner height until the light beam falls within the target area. Check that the slot is parallel to the light path by placing the card at the ends of the burner slot.
- Click on the **Analysis** tab.

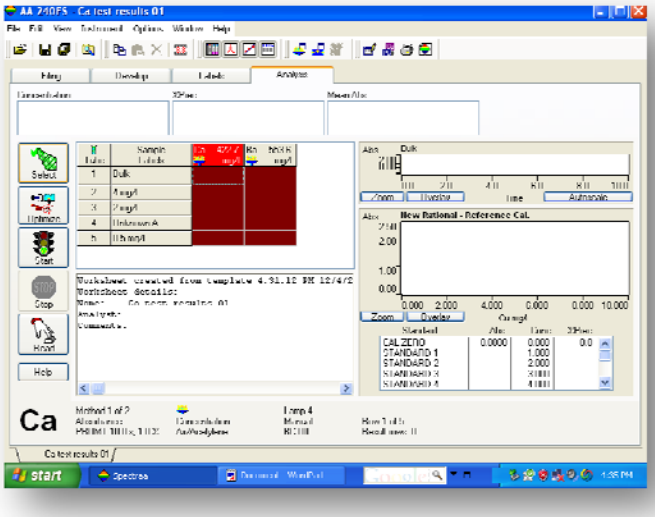

Figure 5. Analysis Tab.

- The **Select** button will allow to highlight the sample to be analyzed; only highlighted samples in the cue will be analyzed.
- The **Optimize** button will allow to **Optimize the lamp** and the signal. In order to optimize the selected lamp, slowly turn one of the lamp adjustment knobs, while watching the lamp signal bar on the display. If the signal decreases, turn the knob in the other direction, until you find the maximum signal. If the HC lamp signal is too small, first check that you have the correct lamp for the current method. If so, press **Enter** to Rescale. This will bring the signal back into range for display. You should also Rescale if the signal becomes too large. Repeat the previous step with the other adjustment knob.
- Turn on the flame by pressing the ignite button on the left bottom part of the instrument.
- To **Optimize the signal** Aspirate the blank and press the **Alt** and **Read** keys together to perform an Instrument Zero. Aspirate a standard solution that will give an absorbance of at least 0.2. Watch the signal bar and adjust the burner height using the outer knob on the burner adjuster to obtain the maximum absorbance, but keep the burner below the light path. Alternate between aspirating the blank and the top standard, making a note of the net absorbance value. When this value stops increasing the burner height is correct. Carefully move the burner horizontally by turning the inner adjustment knob on the burner adjuster. In general, once optimized for maximum sensitivity, this position can be used for all analyses.
- Ensure the pump tubing is placed in the blank solution and press **Start**. The instrument will proceed to perform a calibration by first taking a zero reading and then by performing automatic dilutions from the bulk solution. The software will prompt to indicate which solution to be analyzed based on the sequence labels.
- Alternatively, once the instrument has been calibrated, samples can be analyzed by placing the pump tubing in the solution of interest and pressing the **Read** key.
- The worksheet can be saved in the **Filing** tab.

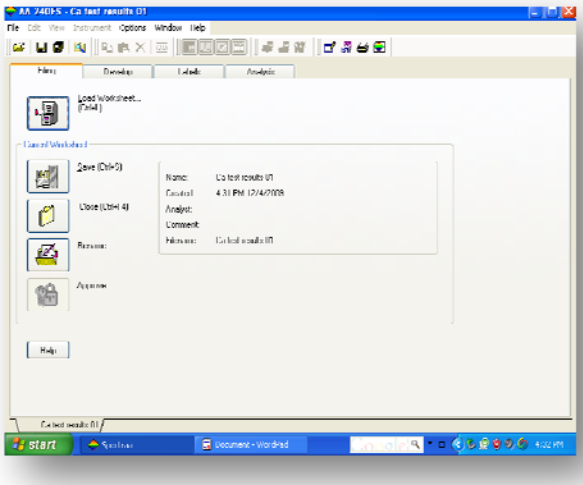

Figure 6. Filing Tab.

- Reports can be generated from the current worksheet or from any other worksheet saved in the system. Access the report window from the **SpectrAA** software shortcut on the desktop.
- The reports window contains four tabs: **Worksheet**, **Select**, **Settings** and **Report**: The **Worksheet** page allows for selection of results to be included in the report. The **Select** page controls which methods and solutions to include in the report. The **Settings** page addresses the report style and content, while the **Report** page allows to manage printing and exporting files as text or PRN.

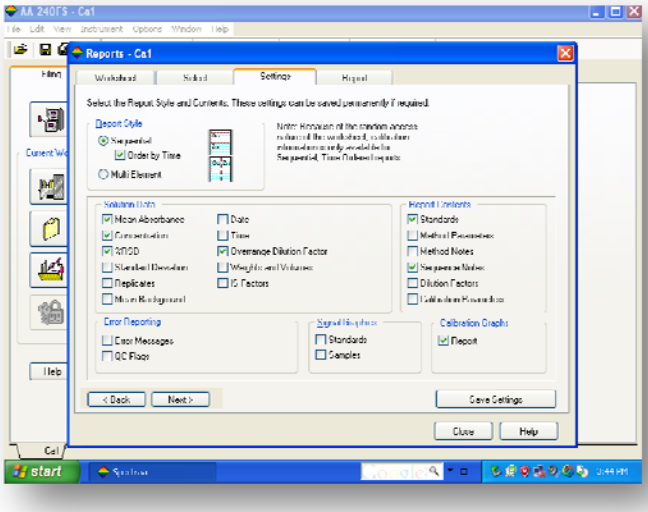

Figure 7. Report Page.

- Turn off the instrument by pressing on the red power off button on the instrument.
- Ensure all gas tanks are turned off and solutions are removed from the SIPS pump.