

AAS Operation Instruction

- (1) Prepare a standard solution and sample solutions

For an accurate quantitative analysis, the concentration of the standard should be close to the estimated elemental concentration of sample. Although the Varian AA240FS with SIPS-10 is able to detect an over range solution and automatically dilute your analyte, less accuracy is expected when a large dilution factor is needed.

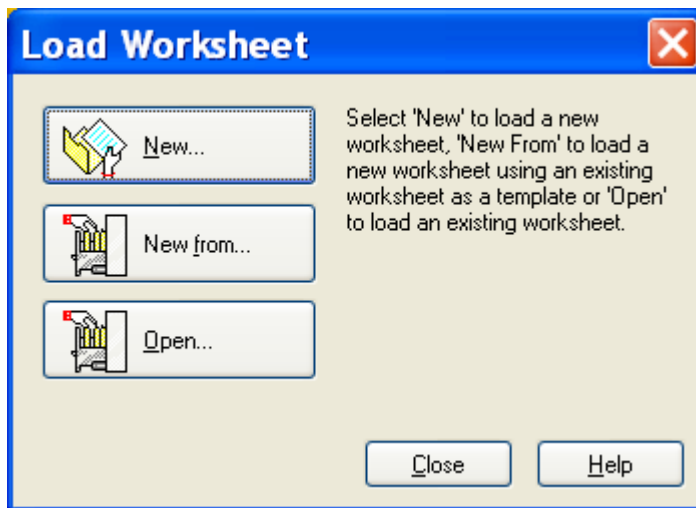
- (2) Fill a beaker with distill water and place the sample solution capillary tube into the water.
- (3) Add distill water (with a 0.01%, mass/volume, concentration of Triton X-100) to the Mariotte vessel.



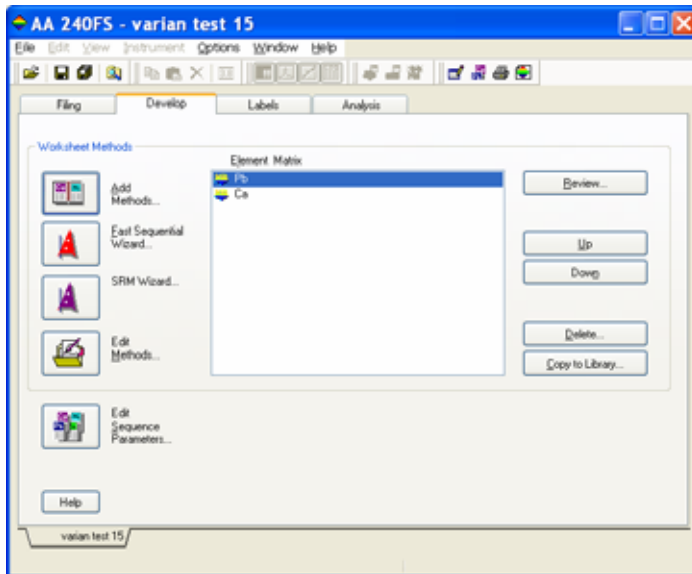
- (4) Place the end of the drain tube inside a vessel to collect the waste.
- (5) Turn on the power of AA240FS.
- (6) Open the compress air valve and set the pressure at 50 psi.
- (7) Open the acetylene valve and set the pressure at 10 psi.
- (8) Run the software SpectrAA.



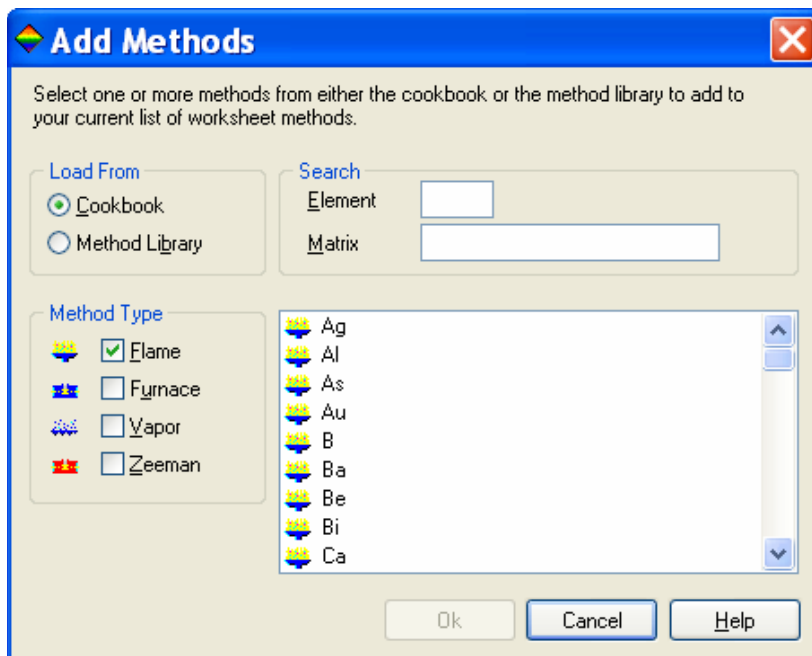
(9) Click on the **Worksheet** and you will see a pop-up window.



(10) Select one of the options to open or create a worksheet. Once the worksheet is opened, you will see a window like this,

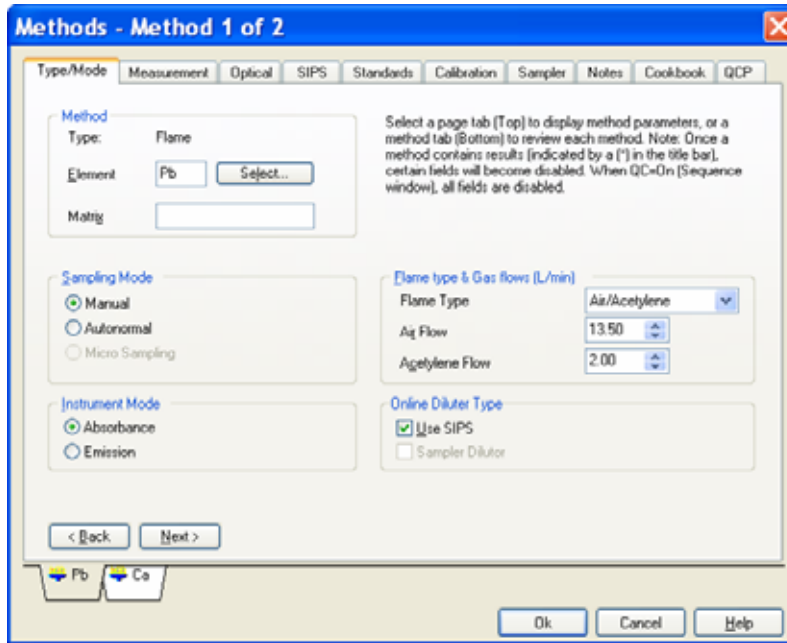


Go to the **Develop** folder. If you are working with a new worksheet, now you need to add a method. Select the element that you are going to test and make sure that the **Flame** box is checked (Our instrument is a flame AAS).



If you are working with a worksheet created from a template, you can also add a method to your worksheet.

- (11) In the **Develop** folder, select the Edit Method to set up the measurement parameters.



Here is a list of parameters for your reference:

Element - Matrix: Pb -
Instrument Type: Flame
Conc. Units: mg/L
Instrument Mode: Absorbance
Sampling Mode: Manual
Calibration Mode: Concentration
Measurement Mode: Integrate
Replicates Standard: 4
Replicates Sample: 4

Expansion Factor: 1.0
Minimum Reading: Disabled
Smoothing: 9 point
Conc. Dec. Places: 2

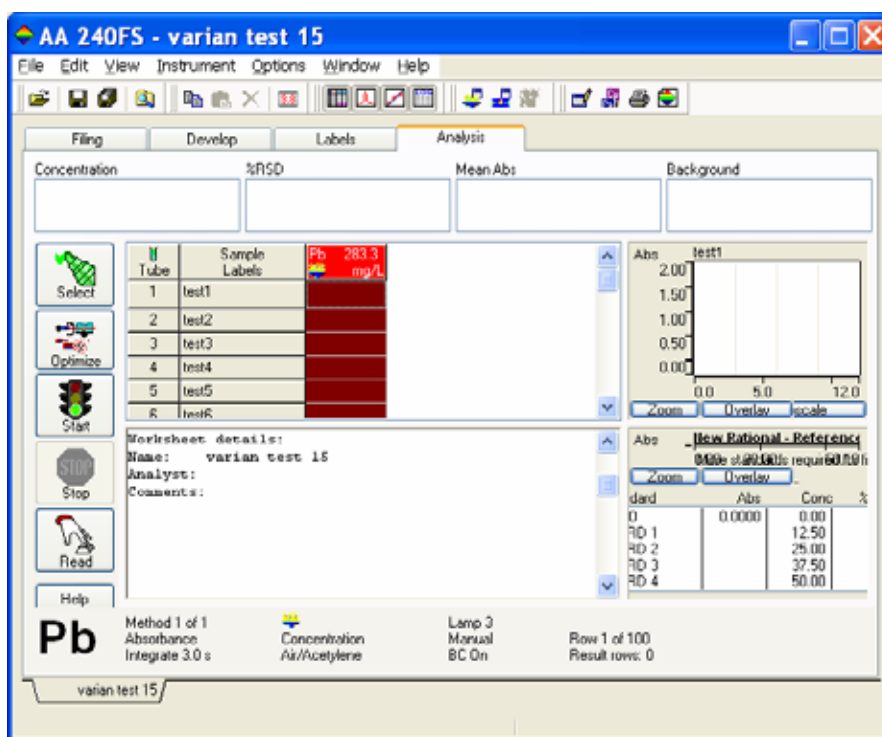
Wavelength: 283.3 nm
Slit Width: 0.5 nm
Gain: 31 %
Lamp Current: 10.0 mA
Lamp Position: 3
Background Correction: BC On

STANDARD 1: 12.50 mg/L
STANDARD 2: 25.00 mg/L
STANDARD 3: 37.50 mg/L
STANDARD 4: 50.00 mg/L
Reslope Rate: 50
Reslope Standard No.: 2
Reslope Lower Limit: 75.0 %
Reslope Upper Limit: 125.0 %
Recalibration Rate: 100
Calibration Algorithm: New Rational
Cal. Lower Limit: 20.0 %
Cal. Upper Limit: 150.0 %
SIPS: On
Neb. Uptake Rate: 5.0 mL/min
Bulk Conc.: 50.00 mg/L
Num SIPS Stds: 4
Initial dilution factor: 2.0000

Measurement Time: 3.0 s
Pre-Read Delay: 3 s
Flame Type: Air/Acetylene
Air Flow: 13.50 L/min
Acetylene Flow: 2.00 L/min
Burner Height: 0.0 mm

Notes: For optimum working condition, choose the wavelength and the slit width according your sample concentration. The Varian Analytical Methods book provides the suggested values of these parameters.

- (12) The **Labels** folder gives you the ability to label each of your tests.
- (13) Go to the **Analysis** window.



- (14) Clean the burner by inserting an index card (provided) into the slit and sliding the card left and right for a few times until it is clean.
- (15) Light up the flame by press the **black** flame button that is located at the lower left corner on the front panel of the instrument.
 Note: (a) You should wait until a stable flame is established before you can make measurement. (b) Press the **red** button next to the flame button will turn off the flame. (c) The flame can be lit up only after you open a worksheet.

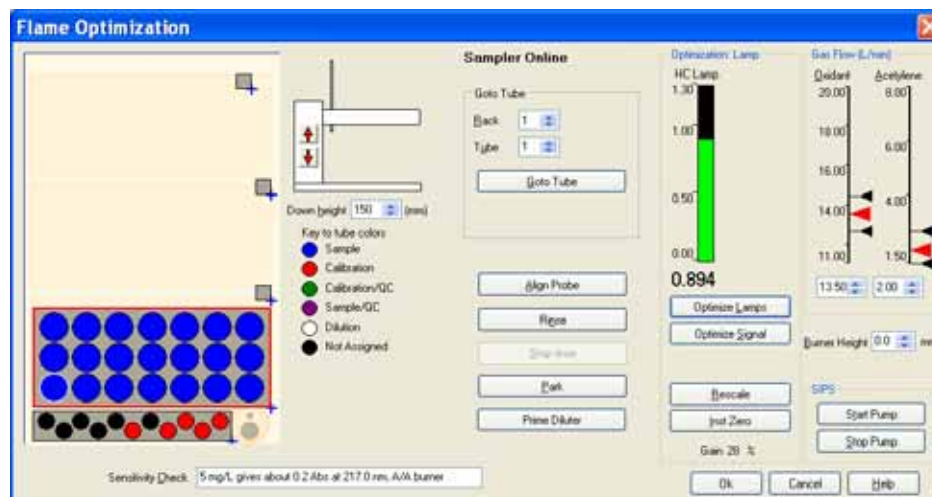


- (16) Click the **Optimize** button and follow the instructions provided by the software to optimize the lamp position. In order to obtain the best signal-to-noise level, try to adjust the lamp position using two screws at the bottom of the lamp base to get the highest HC lamp (the green bar) with the lowest Gain.

The lamp will be turned on at this time if it is not in the **on** status.

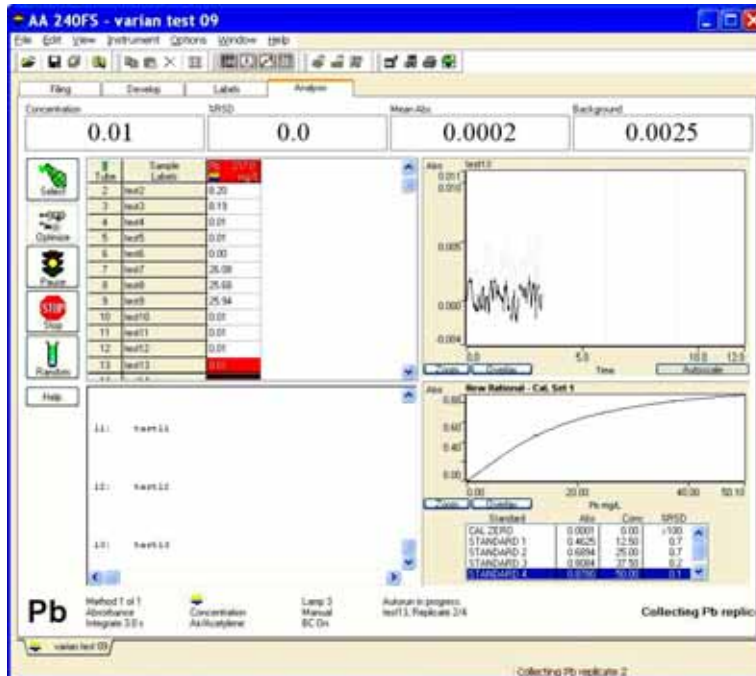
Remember to click the **rescale** button after you adjust the lamp position.

This is needed to reset the gain.





- (17) Adjust the flame height to give you the optimum signal intensity.
- (18) After you have performed the optimization, you can now start your measurement by clicking the **Start** button. The software will give you instructions to tell you how to proceed. Here is the sampling procedures:
 - (a) Firstly, the instrument measures the absorbance of your standard solution to create a calibration. For example, if your standard concentration is $50\mu\text{g/ml}$, and you specify 4 standard counts, the SIPS dilutes your standard to 25 %, 50 %, 75 % and 100 % of your standard concentration to measure the corresponding absorbance and obtain 4 data point to establish a calibration curve.



- (b) Next, you are instructed to place your sample solution for test. If the concentration of your sample solution is below the standard, the result is obtained and shown in the Analysis window. If your sample is in the over range, the SIPS will perform a dilution to make the concentration of the testing analyte within the standard range, then a test is done and the concentration of your sample is recorded. However, if the concentration of your sample is beyond the largest dilution factor that the SIPS can do, the calibration will fail. In this case, an error flag will be displayed in the data cell, for example, “1366.89D”.
- (19) After you have measured all your samples, you should make sure that you save your results. The software will automatically save your worksheet with your results when you close the worksheet or exit the SpectrAA. You can also go to the **Filling** folder to save your worksheet during the measurement. (This will be good for a very unreliable PC that program may crash suddenly).
- (20) The last sample you may want to test is just the distill water only. This is not your real sample but only a maintenance procedure. It just rinses the capillary tube, the liquid trap, the spray chamber and the burner to remove any residual of your last testing sample.
- (21) Now you can turn off the flame by press the **red** button next to the flame button. When you turn off the flame, you may get a warning message on the screen telling you that the SIPS is stopped because the flame is off. This is normal.

- (22) You can view or print your result. To do so, go to the **Window** menu and select the **Report**, then select your worksheet and **Report**, you should see a window like this:

The screenshot shows a window titled "SpectrAA Report." with a toolbar containing icons for print, save, and close. The report content includes a header with the date and time (11:33 AM 10/8/2007) and page number (Page 1). Below the header, there is a section for "Analyst" and "Date started" (2:55 PM 10/3/2007), followed by "Worksheet" (variba test 10) and "Comment". The "Method" is listed as "Pd" and the "Computer name" is "8BLOITCHEMIAA".

The main data table is titled "Method: Pd (Flame)" and has the following columns: Sample ID, Conc. mg/L, % RSD, Mean Abs, and BG Abs. The data rows are as follows:

Sample ID	Conc. mg/L	% RSD	Mean Abs	BG Abs
CALZERO	0.00	7.7	0.0008	-0.0006
	Pe adings:			
	0.0007	0.0008	0.0009	0.0008
STANDARD 1	12.50	0.4	0.2097	0.0010
	Pe adings:			
	0.2110	0.2098	0.2092	0.2089
STANDARD 2	25.00	1.7	0.3818	0.0023
	Pe adings:			
	0.3727	0.3832	0.3830	0.3885
STANDARD 3	37.50	1.1	0.5400	0.0031
	Pe adings:			
	0.5441	0.5463	0.5362	0.5334
STANDARD 4	50.00	1.0	0.6824	0.0033
	Pe adings:			
	0.6737	0.6818	0.6892	0.6848
test1	0.03	74.0	0.0004	-0.0007
	Pe adings:			
	0.0005	0.0007	0.0006	0.0000
test2	0.00	=100	0.0000	-0.0007
	Pe adings:			
	0.0003	-0.0004	-0.0001	0.0001

The status bar at the bottom of the window indicates "0% Page 1 of 2".

- (23) The lamps are not turned off when you close your worksheet. There is also no software control to turn off the lamps except you switch the lamp. The lamp is off when you exit SpectrAA.
- (24) Exit SpectrAA.
- (25) Turn off the power of the instrument.
- (26) Close the Acetylene valve and the Air valve.
- (27) Clean up all your testing solutions.

The detail information about the use of the software and some explanations about the use of this instrument can be found in the on-line help in the SpectrAA.