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.

Load Worksheet	
<u>New from</u>	Select 'New' to load a new worksheet, 'New From' to load a new worksheet using an existing worksheet as a template or 'Open' to load an existing worksheet.
Dpen	<u>C</u> lose <u>H</u> elp

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

◆AA 240FS - varian te	st 15	
Elle Edit View Instrument Q	otons <u>Window</u> Help	
📽 🖬 🖉 🚳 🐘 🕅 X	IIII III I 4 4 1 1 1 1 4 4 5	
Filing Develop	Labels Analysis	
- Wolksheet Methods	Ejement Matrix	
Add Hethods		Beview
East Sequential Weard		Lie Contraction
SRM Wizard		Dowg
Edt Methods		Delete
Edit Sequence Parameters		
Help		
varian test 15		

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).

Add Methods			×
Select one or more methods your current list of worksheel	from either the c t methods.	ookbook or the method library to add to	
Load From	Search		
💿 <u>C</u> ookbook	<u>E</u> lement		
O Method Li <u>b</u> rary	<u>M</u> atrix		
Method Type	 Ag Al As Au B Ba Ba Be Bi Ca 		
		0k Cancel <u>H</u> elp	

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.

pe/Mode Measurement Optical SIPS	Standards Calibration Sampler	Notes Cookbook QCF
Method Type: Flame Element Pb Seject Matrig	Select a page tab (Top) to displa method tab (Bottom) to review ea method contains results (indicate certain lields will become disable window), all fields are disabled.	y method parameters, or a sch method. Note: Ornce a d by a (*) in the title bar), d. When QC=On (Sequence
Sampling Mode Manual Autonormal Micro Sampling	Elame type & Gas flows (L/min) Flame Type Aig Flow Agetylene Flow	Air/Acetylene v 13.50 0 2.00 0
Instrument Mode	Online Diluter Type Use SIPS Sampler Diluter	
< Back Next>		

Here is a list of parameters for your reference:

Element - Matrix: Instrument Type: Conc. Units: Instrument Mode: Sampling Mode: Calibration Mode: Measurement Mod Replicates Standa Replicates Sample	Pb - Flame mg/L Absorban Manual Concentra e: rd:	ice ation Integrate 4 4
Expansion Factor: Minimum Reading Smoothing: Conc. Dec. Places	1.0 Disabled 9 point 2	
Wavelength: Slit Width: Gain: Lamp Current: Lamp Position: Background Corre	283.3 nm 0.5 nm 31 % 10.0 mA 3 c ction:	BC On
STANDARD 1: STANDARD 2: STANDARD 3: STANDARD 4: Reslope Rate: Reslope Standard Reslope Lower Lin Reslope Upper Lin Recalibration Rate Calibration Algorit Cal. Lower Limit:	12.50 mg 25.00 mg 37.50 mg 50.00 mg 50 No.: nit: nit: : hm: 20.0 %	/L /L /L 75.0 % 125.0 % 100 New Rational
Cal. Upper Limit: SIPS: Neb. Uptake Rate: Bulk Conc.: Num SIPS Stds: Initial dilution fact	150.0 % On 5.0 mL/m 50.00 mg 4 tor:	in /L 2.0000

Measurement Time:3.0 sPre-Read Delay:3 sFlame Type:Air/AcetyleneAir Flow:13.50 L/minAcetylene Flow:2.00 L/minBurner 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.

🗢 AA 240	FS - varian t	est 15			
Eile Edit ⊻	iew Instrument (Options Window	Help		
i 🖻 🖬 🖉	😫 🖻 🖻 🗡		300 🕹 🖉 💥	🖬 🖓 谷 💭	
Filing	Develop	Labels	Analysis		
Concentration	*	RSD	Mean Abs	Background	
Select Optimize Start Read	H Samp Tube Label 1 test1 2 test2 3 test3 4 test3 5 test4 5 test5 6 test5 Name: variar Analyst: Comments:	le Pb 2833 s mg/L mg/L		Abs tett1 2.00 1.50 1.00 0	5.0 12.0 eday iscale tional - Reference autoute requie0.tty fi eday . be Conc 2; 00 0.00 12.50 25.00 37:50
Pb varian	Method 1 of 1 Absorbance Integrate 3.0 s test 15	Concentration Air/Acetylene	Lamp 3 Manual BC On	Row 1 of 100 Result rows: 0	

- (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.

	Sampler Online	Optimizations Lamp	Giat Firm L/mm
	Goto Tube	HCLMp 1.30	Quidant Acetylene 20.00 8.80
*	Back 1 (2) Type 1 (2)	1.00	10.00 ¹ 00.01
	(joto Tube	0.50	16.00
Key to Nibe colors		a on	11.00 1.50
Catesson/QC	jälign Probe	0.894	1350 2 200 2
Sample UL Dilator	Rese	Optimize Signal	Barres Height 00 🛫 In
	Doritor Est		
0_0_0_0_000	Prime Dikator	Bescale just Zero	Stat Pump
		Gain 28 1	Stop Pump



- (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 50μ 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:

¢ s	pectrAA Re	port.						×
		► ► 🎒	🛛 🖻	<u>C</u> lose				
	SpectrAA Repo	rt.		11:33 AM 10/8/2	007		Page 1	^
	Analyst Date Started Worksheet Comment Methods Computername Serial Number:	2:55 PM 10/3/2007 Varia 1 de st 10 Po B Elo ITCHEMAA						
		Method: Pb (Flam e))					
	Sample ID CALZEPO		Concing/L 0.00 Peadlogs	% RSD 7.7	Mean Abs 0.0008	BG Abs -0.0006		Ξ
	STA NDA RD 1		0.0007 12.50 Peladings	0.0008 0.4	0.0009 0.2097	0.0008 0.0010		
	STA NDA RD 2		0.2110 25.00 Peladhos	0.2098 1.7	0.2092 0.3818	0.2089 0.0023		
	STA NDA RD 3		0.3121 31.50 Beadlacs	0.3832 1.1	0.3830 0.5400	0.3885 0.0031		
	STA NDA RD 4		0.5441 50.00 Peadlugs	0.5463 1.0	0.5362 0.6824	0.5334 0.0033		
	1e sti		0.6737 0.03 Readings	0.6818 74.0	0.6892 0.0004	0.68 48 -0.0007		
	test2		0.0005 0.00 Readligs	0.0007 >100	0.0005	0.0000 00.00-		
			0.0003	+0.000 ¥	-0.0001	0.0001		~
09	6 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.