

# Brief Outline of Setup and Use Atomic Absorption Spectrophotometer Perkin-Elmer 1100B

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## Preparing to Use the AA Spectrophotometer

### *Conventions*

Key presses are indicated in the text by bold, all capital print (ex. **SETUP**). Keys can either be **Hard Keys**, such as those with printed names on the panel below the screen or **Soft Keys**, which have their names displayed at the bottom of the screen above a set of blank **Hard Keys**. Press the blank key below the screen display to activate the corresponding function. No differentiation is made between **Hard** and **Soft** keys in the text.

All measurements should be made using Absorbance as the technique. The exceptions are measurements for Sodium (Na) and Potassium (K), which may be made using Emission as the technique.

All lamps should be plugged into socket #1 as socket #2 has no power supply.

Almost all measurements will be made using an Air/Acetylene gas mixture. The exceptions are Al, B, Ba and Mo for which Nitrous Oxide (NO) is recommended. Make sure there is NO available before changing the burner. The NO burner has a 5 cm slot while the Air burner has a 10 cm slot. NO is not normally kept available.

## ***Materials***

*Note: Potassium (K) and Sodium (Na) are typically analyzed using Emission analysis. All others use Absorbance.*

Prepare dilutions of standards appropriate to your planned assays.

Prepare a container with de-ionized water for flushing the system.

Prepare solutions of your samples using the same acid/base/solvent solutions/concentrations as the standards.

If desired, dilute your sample solutions so absorbance readings will fall in the linear range of the element to be tested. This is especially important when doing Emission analysis.

If you are doing Emission analysis, ionization is controlled by the addition of an alkali salt to the samples/standards. LaCl (0.1% to 1%) is recommended. Do not use the salt of the element being tested.

## ***Equipment***

*(See pages 8-11, 12, 15, 16 of manual)*

### **For Absorbance Measurements**

Install desired lamps (up to four) into turret assembly. The lamps are located under the cover on the upper left of the machine. Rotate lamp to be used to front, facing you. If the turret will not turn, pull the lamps out to the left slightly so they will clear the window. The turret stops between lamp one and four.

Plug the lamp into HCL 1 socket.

*Note: We use only Hollow Cathode Lamps (HCL) in this machine. They are plugged into socket 1 only. HCL2 is for a secondary lamp to warm-up, but it requires a separate power supply. Lamps should be allowed to warm-up for at least 10 minutes after powering on the machine.*

### **For Emission Measurements**

Set-up as above for absorbance, but choose a lamp matching the standard to be used in burner alignment. The lamp is not used for emission measurements. The excited spectra from the elements in the flame are the light source.

Power on the unit (switch at front left).

After about two minutes, the unit's self-check will be completed. The screen will either display the list of elements or one or more error messages. Reference the errors in Section 15 of the manual to check for solutions.

*Note: Some errors are not problems, as they just indicate missing optional equipment. If the lamp is not plugged in prior to powering-up, the unit will fail its self-check.*

From the element selection screen, enter the desired element number (to match the lamp) and press the **ELEMENT** key.

Reference the “Continuous Mode” operating current range printed on the base of the lamp. Enter the lowest value and press the **LAMP CURRENT** key.

*Note: Use only the values listed for Continuous Mode. Current levels may need to be adjusted upward to maximize energy output. Use the Lamp Log Sheet as a reference. You want to achieve the same energy output as the lamp initially produced with the minimum current value. NEVER exceed the maximum current value printed on the lamp!*

*Note: If a lamp change is done while the power is on, you must set the Lamp Current to zero before unplugging the lamp!*

Press the **FLAME MODE** key, if it is not highlighted already.

## Lamp Optimization

*(See pages 8-7 to 8-10, 18, 7-1.5)*

Press the **SETUP** key.

Once the wavelength value has ramped-up to the preset, a horizontal graph will display.

This represents the current lamp energy level. A numeric value will also display.

Using the two knobs on the turret above the current lamp, adjust the position until a maximal energy level is displayed. If the graph reaches the far right, press the **GAIN** key to re-center the graphic display.

Once the maximum level is achieved using the knobs, carefully rotate the lamp itself by holding the base and gently twisting it about its axis, looking for a maximum energy level.

Continue adjusting the screws and the lamp until no further gains are achieved.

Press the **GAIN** key.

Enter the current lamp current and energy level into the Lamp Log Sheet, with today’s date. If the energy level obtained from maximum current cannot match the initial lamp current, then the lamp needs replaced.

*Note: These adjustments are required every time a lamp is changed.*

## Programming

*Note: Once a program is setup, you can just recall it rather than re-entering it.*

### Entering a New Program

*(See pages 9-1 to 9-12)*

Press the **Prog** key.

Enter the appropriate values for the listed parameters. At the bottom of the screen, calibration standards can be entered. These build a calibration curve, allowing the program to output concentration values instead to raw absorbance values. Check the Analytical Methods Book for specific information.

Follow these basic parameters, unless specific changes need to be made:

Technique:	AA	Lamp Current (mA):	(previously entered)	
Signal Processing:	Hold	Integration Time (sec):	2.0	
Read Delay (sec):	0.0	Printer:	Data Suppl & Print Calib	
Replicates::	3	Oxidant:	Air	
Fuel Flow (L/min)	2.5	Oxidant Flow (L/min)	8.0	
Calibration:	Auto	Standard Units:	mg/L	Sample Units: mg/L
S1:	as needed...	S2:	S3:	
S4:		S5:	S6:	
S7:		S8:	Reslope:	

*Note: Numeric format for S1 indicates the result format for program analysis. S1: X.XXX gives data in form Y.YYY. Enter only as many standards as you wish to use to build the calibration curve.*

## Storing the Program

Press the **USER INDEX** key.

Enter the number for the file to be stored under and press the **STORE** key.

*Note: File number 1 is for the current active program. If a program is recalled and edited, the old version must be deleted before storing it back under the same number. Otherwise, it must be stored under a new number.*

## Recalling a Saved Program

Press the **USER INDEX** key.

Enter the number of the file you wish to recall and press the **RECALL** key.

When Recall stops blinking, the file has been recalled to position 1, the active file.

## Burner Alignment, Nebulizer and Fuel Adjustments

*Note: Generally, alignment is only required when the burner and/or nebulizer has been replaced or has been adjusted improperly. Once you have set the optimal alignments/adjustments, you should not need to change them. Skip to Flame Ignition if alignment is not needed.*

### Burner Alignment

*(See pages 9-44 to 9-46)*

#### *Coarse Adjustment*

Press the **CONT** key to enter continuous scanning mode.

Adjust the burner downward, below the light path, using the vertical control knob on the lower left of the assembly.

Now, adjust the burner upward until an increase in absorbance is indicated on the screen.

Readjust the burner to the point just before the increase occurs, then lower it another quarter turn.

#### *Fine Adjustment*

Ignite the burner flame as described in the Flame Ignition section (after Alignment section).

Aspirate a blank solution with the capillary tube.

Press the **AUTO ZERO** key. The trace should drop to the base line.

Aspirate a reference solution of an element (preferably, the one you will be using).

*Note: The solution concentration must be in the linear range for that element and preferably near an absorbance of 0.2.*

Adjust the horizontal, vertical, and rotational control knobs (lower right, left and upper right, respectively) to maximize the absorbance reading on the screen. Do not adjust the vertical control to the point where the burner head enters the light beam.

If required, adjust the nebulizer (as described in the next section) to maximize the absorbance reading as well.

Alternate between the burner controls and the nebulizer adjustments until no further gains are achieved.

If required, adjust the fuel flow rates (as described in the next section after nebulizer adjustments) as well.

*Note: Fuel changes are generally only required if different flame characteristics are called for by the element.*

## Nebulizer Adjustment

*(See pages 9-46, 47)*

*Note: Only adjust the nebulizer while using an Air/Acetylene flame. NEVER with Nitrous Oxide!*

*Nebulizer adjustment are only required when the assembly has been dismantled for cleaning or to optimize when changing solvents. Do this in conjunction with burner alignment.*

Choose a reference solution requiring a blue (oxidizing) flame and wavelength above 250 nm (Cu, Mg, Ni, Pb).

Do not use Ca, Sr, Ba, Pd, Pt, Rh, Ru, Au, Ir or others whose radiation absorption is affected by air/fuel ratios.

Aspirate the reference solution (again, concentration yielding an absorbance of about 0.2).

Loosen the locking ring on the nebulizer port, where the capillary tubing attaches. Turn the knurled cap counter clockwise until bubbles appear from the tubing in the solution.

Now, turn the cap clockwise until a maximal absorbance reading is attained.

*Note: The first peak is not necessarily the maximal one. Run through the range to verify the largest absorbance reading.*

Snug the locking ring against the cap to prevent it from moving.

## Gas Flow Adjustments

*(See page 9-47)*

*Note: Do this in conjunction with the burner alignment procedure above and/or to adjust flame properties (oxidizing/reducing, lean/rich fuel mixtures, color).*

Press the **ATOM CONTR** key.

Aspirate a reference solution.

Using the **C<sub>2</sub>H<sub>2</sub>←**, **C<sub>2</sub>H<sub>2</sub>→**, **OXIDANT←**, **OXIDANT→** keys to adjust the flow rates up or down.

Make incremental changes to adjust the flame characteristics and/or maximize to absorbance reading.

## Flame Ignition

*(See pages 9-39 to 9-43)*

Turn on the Air and Acetylene gasses at the tanks.

*Note: Incoming pressures from the tanks should be as follows: Air= 58 PSI, Acetylene= 15 PSI, Nitrous Oxide (if equipped)=58 PSI.*

Do not adjust any valves except the main tank valve unless outlet pressures are incorrect.

*Note: Change the Acetylene tank when the pressure reaches 85 PSI to avoid drawing acetone into the burner regulator.*

Verify all items in the following checklist **BEFORE** ignition:

- Gas inlet pressure to unit is within range (see above).
- Do not use "Breathing Air" or anything with excessive oxygen levels as oxidant.
- Proper burner head gasket is in place.
  - Acidic/Basic solution requires the rubber gasket.
  - Organic solvents require the cork ring.
- Correct burner head is installed (NO vs. Air oxidant).
- Burner head slot is clean and free of deposits.
- Use a corrosion-resistant nebulizer if necessary (See manual section 16.2).

- Fuel and nebulizer line securely connected to burner assembly.
- Siphon interlock filled with water.
- Float in interlock moving freely.
- Drain tubing attached and free of kinks.
- Neck of non-glass waste jug is open.
- Fluid level in jug is visible.
- No air bubbles in U-loop of siphon interlock.
- Door panel of burner chamber is closed.

Place the aspiration capillary tube into a container of de-ionized water.  
Press the **FLAME-ON/OFF** key.

*Note: The unit will shut down if no water is in the trap. It may shutdown and respond with an error message as well. If first try at ignition fails without error, try again.*

**NEVER** leave the flame burning while unattended!

**ALWAYS** aspirate some solution (or water) while flame is burning!

Keep all containers of flammable s away from the flame. If using volatile and/or flammable solutions, keep containers closed/covered when not in use. Cover container with parafilm while aspirating.

## Operating the AA Spectrophotometer

### *Running a Program*

*(See pages 9-17 to 9-30, 9-48 to 9-53)*

#### **For Emission Measurements Only**

Continue to aspirate the blank or de-ionized water.

Press the **ELEMENT SELECT** key.

Enter 0 and press the **LAMP CURRENT** key to turn off the lamp (not needed any longer).

Lamp is off when Lamp Current is no longer highlighted.

Enter element number and press the **ELEMENT** key (19 for **K**, 11 for **Na**).

Press the **Prog** key.

Press the **EMISSION** key to select Emission analysis.

Recall or create the appropriate Emission program for the analysis. Use the steps described above in the Programming section. Be sure to use **EMISSION** as the technique.

Verify that the Lamp Current parameter is set to zero.

Aspirate the highest selected standard concentration (you **will** use a lot of solution).

Press the **SETUP** key.

Check for proper element, wavelength and slit width settings, as per the standard conditions for the selected element.

While the wavelength is reset, observe changes in the bar graph, indicating varying levels of emission at that point.

Press the **RUN** key.  
Flame Run Mode screen will be displayed.

## Printer On

Turn on the power to the printer.  
Press the **PRINT** key. Press the **LQ 850** key.  
If the screen **PRINT** key is not highlighted, press it now.  
Press **MAIN KEYS** key to continue.

## Calibration

Aspirate an appropriate blank solution and press the **AUTO ZERO** key.  
Depending on the number of standards set in the program, press the **STDS 1-4** and **STDS 5-8** keys as appropriate.  
Aspirate the first standard, then press the **STD 1** key.  
Repeat for each standard you have to measure.  
When calibration is finished, a curve and its data will be printed on the printer.

## Measurements

When calibration is finished, prepare your sample solutions.  
Aspirate your sample and press the **READ** key.  
All readings will now be converted to concentrations according to the calibration that was just completed.  
If replicates were entered in the program, multiple reads will be done on each sample and the results with statistics will be displayed when sample is finished.  
If printer is on, results should also print on printer.  
Between samples, always wipe off the capillary tubing and aspirate de-ionized water through the nebulizer to prevent deposits.  
Repeat for each sample.

*Note: Remember to keep all concentrations in the linear range of detection when doing Emission analysis.*

## *Shutting Down...*

### The Lamps

Press the **SETUP** key.  
Enter 0 and press the **LAMP CURRENT** key.  
The graph display should show no energy levels.  
Unplug the current lamp.  
If the lamp is to be changed, rotate the new lamp into place and plug it in.  
Resume with the Lamp Optimization section above.

### The Flame

When finished with your measurements or adjustments, aspirate de-ionized water for several minutes.  
Press the **FLAME ON/OFF** key to extinguish the flame.  
If no further flame use will occur, then continue shutdown with the next section; otherwise repeat the appropriate steps in the Flame Ignition section above when ready to re-ignite.

## The Machine

Complete the two shutdown procedures above.  
Leave the lamps unplugged.

Turn off the fuel and oxidant gasses at the tanks.  
Press the **ATOM CONTROL** key.

*(See pages 9-31, 32)*

Press the **CHECK GASSES** key.  
Press the **CHECK F&O** key. You will hear gas flow as the remaining line pressure is released. When cycle is complete, repeat key press two additional times.  
Check the gas tank gauges to verify that line pressure is zero, especially the acetylene line.

Turn the power to the printer off.  
Turn off the main power switch, on the lower left of the unit.

Clean up the work area. Remove all samples and solutions. Put away all standards.  
Check and clean burner head. Use the thin, flat metal bar across the top of the head and in the slot to remove any deposits.  
Use the fine copper wire to carefully ream out the nebulizer capillary tubing and orifice.  
Take care not to bend or kink the wire or tubing.

## Maintenance

Contact the Lab Manager with any problems and/or concerns regarding this equipment and related issues.

Clean and wash down parts with soap and water monthly. Rinse thoroughly in de-ionized water.

Remake standards as needed. Standards in 1% nitric acid are good for at least 30 days.  
A 1% to 1.5% nitric or hydrochloric acid solution is most common for standard preparations.  
Keep sulfuric and perchloric acid concentrations between 0.1% and 0.5% to control corrosivity and viscosity problems.  
Always use the same acids and concentrations in the blanks and standards as was used in the sample preparations.

*Note: Use plastic bottles for standards. Glass is too porous.  
See information regarding standards in section 7.5 of the Analytical Methods Book.*

If organics have been used, check and/or change burner o-ring.

After long periods of inactivity:

Keep the siphon interlock full of water and free of contamination (molds, etc.). Remove and clean as needed.  
Remove and clean/lubricate the nebulizer.  
Re-align the burner and optimize the nebulizer as needed.