

Directions for Using the Buck 211 Atomic Absorption Spectrometer (AA)

Setting up the AA software:

1. Put on your safety goggles and a pair of disposable gloves! Next, push in the square red power button (on the right side of machine) on the AA. You may assume that the Pb hollow cathode lamp has already been installed and properly aligned.
2. On the right side of the machine are two knobs. One controls the slit width, and can only be set to one of three numbers. Set it to 7 Å. The other knob controls the AA's detector wavelength. Pb atoms emit and absorb most intensely at ~283 nm. Set the dial to this number. (Note that the wavelength control knob actually adjusts the angle of the diffraction grating in the machine). Our goal is to "aim" the 283-nm light directly onto the photomultiplier tube (PMT).
3. At the top right corner of the screen, below "Lamp 1", it should read "D2 Bkg Comp On". If it reads "off" instead, press the **BKGND** button on the machine.

Do the following three steps before you retrieve your standards and analyte solutions—you want to maximize the warm-up time for the instrument.

4. Press the **SEL** button on the front control panel (bottom right key). You may have to wait a moment for the machine to warm up. The top right of the screen should then say LAMP 1, 2, or 3. Pressing the **SEL** button again will change the lamp number. Continue to press **SEL** until the screen reads LAMP 1. Then press **ESC**.
5. Next, you will maximize the light intensity hitting the PMT. Do this by pressing the **ALIGN** button on the front control panel. There will be 2 bars, each over a number line, and above that a value labeled "Energy" and "Bkg Energy". The goal is to make the "Sample" bar read as high a positive value as possible, and thus make the "Energy" number as high as possible (they increase in tandem). To do this, turn the wavelength knob on the side of the machine in either direction until the energy is at its maximum. Do not be surprised if the optimal position is a few nm different from the nominal λ_{\max} – the wavelength dial is only approximate. In your notebook, record the wavelength and energy readings obtained once the energy has been maximized.
6. Press the **A/Z** button to autozero the machine at its maximal energy. Wait until the screen returns to the main, initial "Active Analysis" screen.

Preparing the AA for use:

7. Your standards and analyte solutions should be retrieved before moving beyond this step. Immerse the AA's thin Teflon aspirator tube in a 250-mL Erlenmeyer flask full of distilled water. Keep this tube immersed in liquid while the flame is on – that is, if the AA sucks all the water out of this flask, be sure to re-fill it within a few seconds; don't let the flame continue to burn for very long (15 seconds) without aspirating a liquid.
8. Open the yellow air jet along the wall to your left, which is connected to a filter system via a braided nylon hose. (It says "Apollo" on it, and is open when the handle points toward the back wall.) Confirm that the black and red gauge connected to the filter in the air line (above and to the right) indicates that the line pressure is between 60 and 70 psig.

9. Open the main valve (directly on top of the cylinder) of the acetylene cylinder behind the AA bench completely. **The cylinder should not be used if the cylinder pressure (right-hand gauge) has dropped below 50 psig (lb/in²)**, as too much acetone will be extracted (acetylene is dissolved in acetone to prevent explosions). [If the pressure is near 50 psig, please inform Professor Kuwata]. The line pressure (left dial) should be between 13 and 14 psig (and definitely not more than 15 psi). Unscrewing the large black knob of the regulator will lower the line pressure, but only if you press in the white **AIR** button on the AA to release some of the gas pressure.
10. On the left part of the machine, press and hold **AIR** (bottom right button). The columns for air (“oxidant”) and acetylene (“fuel”) should both read near 5 on the flow tube bobs below. If the “fuel” is too low, adjust with the “Fuel Adjust” knob below. Before igniting the flame, wait 1 minute after pressing **AIR**.
11. To ignite the flame, press and hold **ON** for 5-8 seconds while repeatedly pressing the trigger for the old red and white ignitor above the burner head. **Or, use the manual ignitor, holding its tip over the burner slot. (If you ever need to extinguish the flame quickly, press OFF at any time.)** Water should begin to be aspirated.
12. Make sure you are always aspirating some liquid whenever the flame is on. Otherwise you risk overheating and damaging the burner. Re-fill the 250 mL flask with deionized water as needed. The nearest DI water tap is at the sink BEHIND you!
13. Check that the flow rates of “air” remains close to 5 (as measured by the flow tube bob), and turn the "FUEL ADJUST" knob to lower the fuel flow rate to 3. The flame should go from being bright yellow to a dull orange, remaining bright blue at the very bottom.

General measurement procedure:

14. If the absorbance reading on the screen is more than ± 0.003 away from zero, press the **A/Z** button again.
15. *Do not aspirate standards or samples before the machine has aspirated pure water for 5 minutes.* Aspirate your 20-ppm Pb standard by quickly moving the aspirator tube from the water to the standard. Press **READ** to integrate the value for the absorbance. The screen should then display a constant absorbance value. Then aspirate water for at least a minute.
16. Repeat this procedure for each of your other standards, and each of your analyte solutions. You should cycle through all of the solutions at least three times. Re-zero the display with the blank solution (distilled water) after each solution.
17. Finally, make nine measurements on your 1-ppm solution. Aspirate water (and re-zero if necessary) after every three measurements.

Shutdown procedure:

18. Run distilled water through the aspirator for a minute to rinse the burner head.
19. Stop the acetylene flow by pressing **OFF** on the left part of the machine.
20. Close the main valve (right side) on the acetylene cylinder completely.
21. Close the yellow “air” supply valve to the left of the AA.
Press the AIR button until the excess air and acetylene in the instrument has been vented.
22. Turn off the spectrometer by pressing the square red button on the right side of the AA.
23. Clean up after yourself! Remove all of your solutions, and clean up any spills.