

Operation of Haverford College Atomic Absorption Spectrometer (Buck Scientific Model 211 AA)



1. Press the red button on the right side panel of the machine to turn on the lamp as well as the instrument's built in computer. Wait 20 minutes before data collection for the lamp to warm up. If you need to change lamp or if the AA has not been used for a few months, align the wavelength of the lamp (see pages 16 to 17 of the manual). You may also need to align the lamp position after you change lamp.



2. Make sure the vent is turned on by turning the lever so that it points down (see picture).



Also pull out the sheet of metal blocking the tube.

3. To ignite the Air/Acetylene flame (p21 of manual),



- Open the valve on top of the acetylene tank (not the N₂O). And don't touch anything else.



- Check that the house air is 60psi:

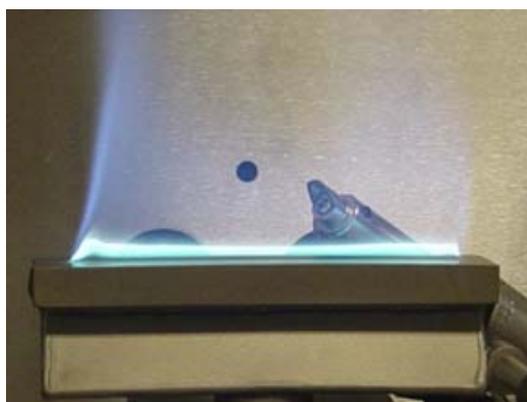


- Check that acetylene tank pressure is set to 13psi.

- Press and hold the [air] button on the left panel to see if you have at least a flow of 5 for both the air and acetylene. If not, adjust the flow of fuel using the valve on the left panel. Without enough fuel the flame will not ignite. Release the [air] button when finished. If you need to shut down the flame at any time, press the [off] button on the left panel. The only lights on now should be the N₂O Interlock light.



- Press and hold [on] button for a few seconds until the flame ignites then release the button. Now both the N₂O Interlock light as well as the Flame Sensor light should be on. If the flame does not ignite, recheck the gas flows and try again.



- For most experiments, use the valve on the left panel to adjust fuel so that it is around 3.5. You can also adjust the flame so that the flame is more concentrated coming out of the burner head. This is an example of a bad flame (acetylene flow too high – gives flickering flame of uneven height):



- This is an example of a better flame:

- For some experiments it is possible that you may require a higher flow. (see manual page 21)
- Wait 5 minutes after igniting the flame before data collection.

ACTIVE ANALYSIS		LAMP 1	
Name:	Co-D2-240.7-1ib3	D2 Bkg	Comp Off
Lamp:	Co Buck Sci		
Meth:	Air/Acet Abs	Wvl:	240.7 nm
Curr:	4.5mA	Slit:	0.2nm
		Pat:	316.3V
		Intgr:	3.0S
Time:	3:46 PM	Wed	Jun 8, 2005
Smp Energy:	3.298		
Abs:	0.006		
Abs:	0.006		
STANDBY ANALYSES			
As-D2-193.7-1ib3	Flame	D2 Bkg	Lamp 2
Ba-D2-553.6-1ib3	Flame	No Bkg	Lamp 3

4. From the Active Analysis Screen shown, press [Cal]

FLAME CALIBRATION FORM			
Cup	Name	Conc	Abs
151	Std1-Blank	Autozero	
152	Std2-Max	30.000	0.000000
153	Std3	20.000	0.000000
154	Std4	10.000	0.000000
0			
0			
0			
0			
0			
Smp Energy:	3.296		
Abs:	0.000		
<ESC>: exit <HELP> <CNTLS>: Cal Cntls			

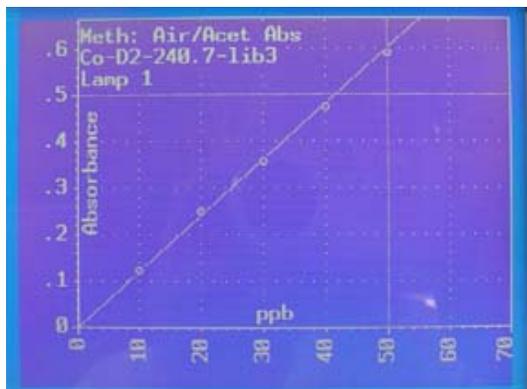
5. In the Flame Calibration Form shown, enter the concentrations of your standards into the CONC column starting with cup 152¹, the most concentrated standard solution. You can move across the row with the [sel] button and up and down the column with the corresponding arrow buttons. You can add more calibration standards by entering the next cup number into the CUP column of an empty row and press [enter]. You can also delete a row by holding [upper case] button while hitting [del] then [enter]. By default the first cup is 151 and is a blank solution. You can only add up to cup 156, to add more see page 25 of the manual.

FLAME CALIBRATION FORM			
Cup	Name	Conc	Abs
151	Std1-Blank	Autozero	
152	Std2-Max	50.000	0.650000
153	Std3	40.000	0.561000
154	Std4	30.000	0.460000
155	Std5	20.000	0.350000
156	Std6	10.100	0.175000
0			
0			
0			
Smp Energy:	3.334	Bkg Energy:	3.635
Abs:	-0.003	Bkg Abs:	-0.029
<ESC>: exit <HELP> <CNTLS>: Cal Cntls			

6. Without exiting from the Flame Calibration Form, press [Start]. The screen instruction will prompt you to insert the capillary tube into cup 151, the blank sample (usually de-ionized water). Wait at least 5 seconds after inserting the tube into each solution—this is how long it takes for the sample to go through the tube into the flame. After about nine to ten seconds, the reading usually stabilizes to the 2nd decimal. Once it is reasonably stable, press [enter] once the absorbance stabilizes; the machine will then average the absorbance readings over the next 3 seconds and subtract this value from the subsequent readings.

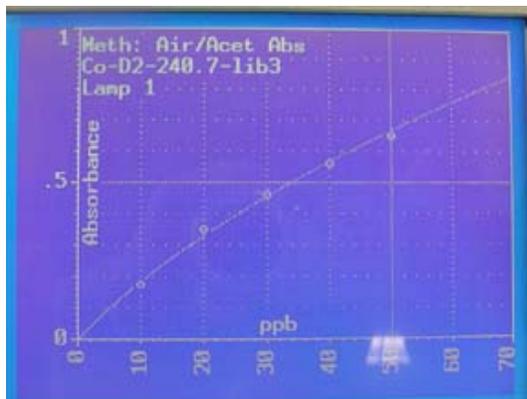
¹ The software assumes we have an autosampler, which we don't. So ignore the "cup" language.

The machine will then prompt you to insert the tube into your first standard solution. Repeat the process as prompted by the screen instruction – by default it will prompt you to read your calibration sample two times, so you will have to hit [enter] twice after reading is stable in order to get two, three-second readings.



7. Once finished, press [cal], then [yes], then [enter] to try a linear fit to your calibration data. A good calibration graph has all the points going through the curve, as shown below for a linear calibration graph:

If the data points do not all lie on the calibration curve, type [esc], [cal], and change “Degree” to “2nd”. This will curve fit your calibration data to a second degree polynomial.



8. Press [cal] and follow the instruction on the screen to view graph of your calibration. To quit from the graph window, press [esc]. You can adjust how you want to curve fit your data again by pressing the [cntrl] button. This is an example of a good quadratic fit.

9. Once satisfied, exit the Flame Calibration Form by pressing [esc]. Insert the capillary tube into your samples to take readings. Like the calibration standards, the readings will tend to fluctuate after you put it into the sample, but will tend to stabilize to the ones digit after about 9 seconds. When the reading stabilizes, press [READ]. The machine will integrate over a 3 second period and output the reading. Allow at least 5 seconds after inserting the tube before reading the absorbance. Also put your tube into de-ionize water for a few seconds after each reading and make sure to wipe the tube with Kim-Wipe afterwards. The beam strength may fluctuate, so you may want to put the tube in de-ionize water and press [A/Z] every five minutes to auto zero.



10. To adjust for the number of digits in the output, press [cntrl] and select the “Report and Display Control” menu. From there, adjust for the decimal places in the concentration or absorbance output accordingly.

11. Write the absorbance and concentration in your notebook.

12. To turn off the flame press [off] to extinguish the flame. Close the acetylene tank valve and do not touch the pressure regulators. Press [air] until the fuel and air level both drops down to zero. Turn off the lamp and computer by pressing the red button to the right side of the AAS.