Using the atomic absorption spectrophotometer (PerkinElmer PinAAcle 900T)
The atomic absorption spectrophotometer is used to determine the concentration of an element in the sample. It converts a liquid sample to a gas by fragmenting the sample into small drops (atomize) and then feeding it into a flame. The spectrometer uses the radiation wavelengths absorbed and emitted by different atoms to measure the concentration the element.
Reference: Analytical Methods for AA Spectroscopy

Setting up for a run
1. Turn on AA by flipping on the green switch. Make sure that aspirator tube is out of any solution (tube should be aspirating air).
2. Open Syngistix software
3. Open correct method file and enter calibration curve concentrations
    a. Click Analysis > Method
    b. In the method editor that is now open, click Calibrations > Standard Concentrations
    c. Enter both the ID and actual concentration of each of your standards
4. Change out the burnerhead if necessary with the magnetic strip facing towards the back. You must push down the white button on the nebulizer assembly and pull the grey lever completely to the forward position to put burnerhead in place

Entering calibration curve
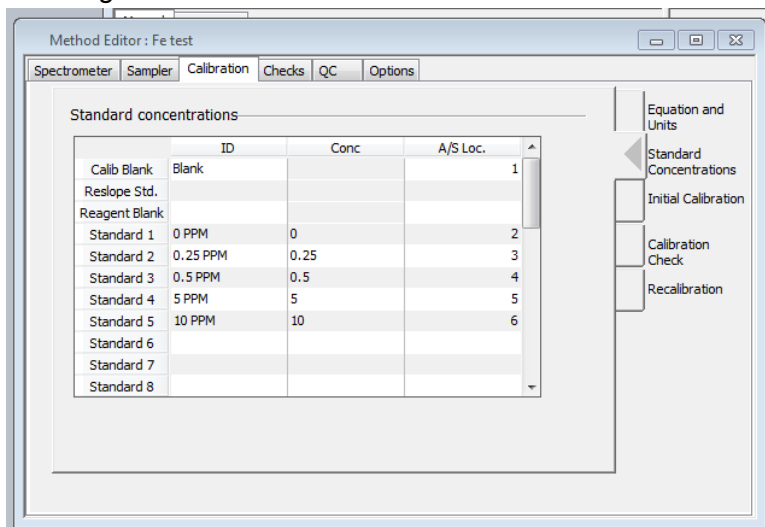
Turning on the flame
1. Switch on the air compressor and turn on the gas by turning the knob on the top of the acetylene tank counterclockwise
2. Turn on flame control (Instrument > Flame Control) in Syngistix and turn on flame
3. Allow burnerhead to warm up on air for a few minutes
4. If necessary for your analysis, switch flame to nitrous oxide/acetylene in the flame controls (must click "Apply"). Allow burnerhead to warm up for another 15 minutes
5. ***SAFETY NOTE: Always wear the welder's glasses when looking directly at the flame.*** Look at the flame. There should be a red "feather" in the middle of the flame. If

the feather is shorter than 1.5 inches vertically, increase the acetylene flow rate to about 8L/minute (must click "Apply" after changing flow rate).

Flame control
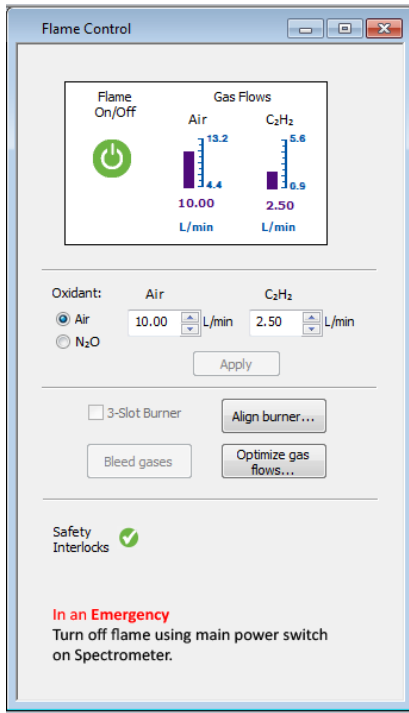
Using the align burner wizard
1. Open continuous graphics (Instrument > continuous graphics).
    a. While aspirating air, press the button to "Auto Zero Graph"
2. Use burner alignment wizard to automatically adjust vertical and horizontal direction of burner. Follow directions on wizard. You may need to use the burnerhead key that fits in the front hole on the burnerhead to slowly rotate the burnerhead until your 50 ppm standard reads at ~ 0.2 nm in continuous graphics.
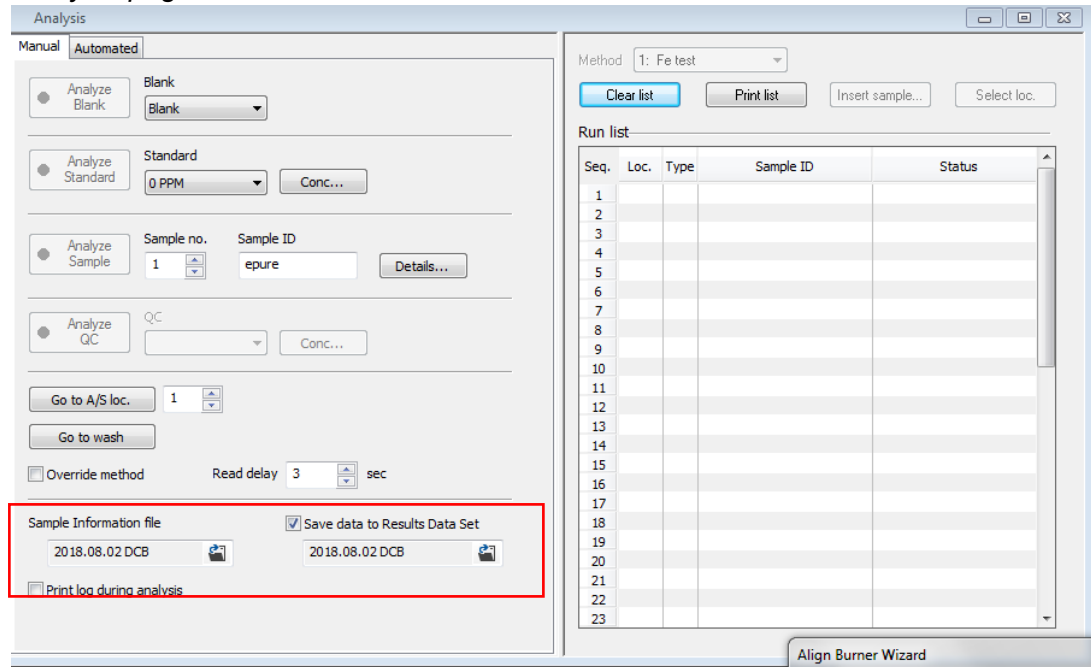
Continuous graphics
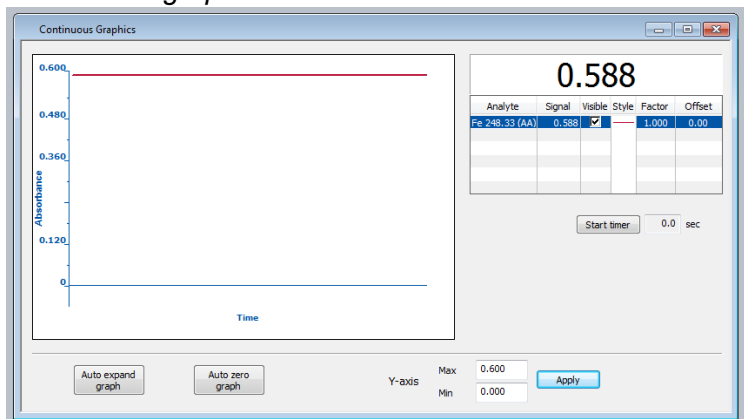
Entering sample data
1. Go to Analysis > Sample Info. You can copy/paste your sample IDs into the software here.

2. You can set the results output file name simply by typing in a new name in the box under "Save to results data set"

Analysis page

Running standard curve
1. Click Results > Results to open results sheet. Then go to Analysis > Analysis.
2. Stick aspirator tube in deionized water and click "Analyze blank" in the Analysis tab. Allow machine to run all three reps. Wait for auto-zero performed readout in the results sheet.
3. <u>Now you are ready to run your standard curve</u>. Stick aspirator tube in first standard and click "Analyze Standard." **Clean tube by aspirating a small amount of the 5% nitric acid for 3-5 seconds between samples, allowing flame to return to initial size and color between samples.** Run the rest of standards and click Results > Calib. Calibration coefficient should be 0.995 or better.
4. In the analysis tab run a matrix blank after your standard curve to double check calibration. You may insert it as a sample. Click "Analyze Sample.

Analyzing samples
1. Now you are ready to analyze your samples.
   a. Analysis > Sample info > Paste sample table
   b. Analysis > Check sample info name and save location > change file save path if incorrect
   c. Repeat step 16 and clean aspirator tube by aspirating a small amount of the 5% nitric acid for 3-5 seconds between samples.
   d. Check residuals in results tab. If they are higher than 20%, rerun the sample. If any sample is higher than your calibration curve, make a note and dilute that sample to rerun later.
2. Periodically run a "mini calibration curve" to check for instrument drift by analyzing a blank and two samples which represent the range of your samples as an unknown (~ every 15 samples).

<u>Turning off the AA</u>
1. If you used nitrous oxide, when you are done running samples, switch flame back to air/acetylene in Flame Control (hit "Apply"). Allow to run for about 20 seconds and then switch flame off completely.
2. Close gas tanks (turn tank knob all the way clockwise), turn off air compressor, and unplug nitrous oxide regulator.
3. In flame controls hit "Bleed gases." ***Note: this is an important safety step as it will bleed all gas remaining in the machine tubes. Do not bleed gases unless gas tanks are closed and air compressor is off.***
4. Turn off Syngistix. Make sure to accept to "Turn off al lamps"
5. Turn off AA.

<u>Getting data</u>
1. Open Data Manager software on the desktop.
2. Click "Export."
3. In data export wizard click "Use existing Design" and open up the file titled
4. "AA_report.xptx"
5. Go through the rest of the data export wizard and select desired report output parameters.
6. Save file.

TROUBLESHOOTING
- Cannot run samples or edit method at the same time as "continuous graphics" window is open
- Proboscis is aspirating water but the stream from the drain drips and is not steady – aspirate coffee for 5 minutes and attempt again
- Burnerhead not locked – jimmy the burner head around, make sure that grey lever is all the way forward.
- Dirty burnerhead – soak burnerhead upside down (DO NOT submerge magnetic strip - only top of the burnerhead) in 10% nitric acid for 30 mins. Rinse thoroughly with deionized water and let air dry.
- Uneven flame – there may be buildup on the burnerhead. Use graphite stick to clean burnerhead with flame on.
- Flame won't light – use Bunsen lighter to manually light flame.
- Drain Interlock – unscrew the draining lock and pour DIW down the drain tube
- Clog in nebulizer – nebulizer needs "oiling", aspirate coffee
- No air pressure error – using a flathead screwdriver, loosen the hose clamp on the waste drain line. Pull the off of the machine and pour a stream of deionized water down the line. Reconnect the line to the machine. Error message should disappear.