AAS SOP - Flame

Start-up

1. Turn on gas tanks** outside. Turn on the heater for N2O gas. Must use safety goggles when N2O flame is in use.
2. Check Compressed air pressure. It must be at **8 bar** on compressor gauge
3. Open respective gas valves.
4. Turn on Hood (#20)
5. Turn on the machine (when gases reach set pressure)
6. Turn on software (Click on Start Menu on bottom left side of screen - Click on Computer - Local Disk - Program Files (x86) - PerkinElmer- WinLab32-AA)

** Acetylene delivery pressure must be between 0.9-1.0 bar. Internal pressure must be at 4 bar and never above to avoid explosion.

** All gas tanks pressures should be at the black mark labeled on the gauges.

Troubleshooting Flame

1. If "Interlock: No Air pressure Message" appears
    a) Make sure all gases used have appropriate delivery pressure
    b) Make sure drain bottle is not full. Empty it if it is
    c) Run 500 ml of DI water through hose into the drain bottle
    d) If above steps do not work, readjust delivery pressure on gauge after the filer
        i. Lower burgundy cap
        ii. Turn left or right to go to the black line as marked
        iii. Press cap back up to lock

Manual Testing Set-up

1. Install appropriate lamp(s) in chamber
    - Lamp check
    a) Click "lamp" tab on top of screen (lamps are automatically detected)
    b) Click on the "On" circle on left side. Check the "desired current" in milliamps. Make sure it is not beyond the max allowed (25 for Cu)
    c) Click on "Lamp 1". Wait 10 minutes for energy reading to stabilize

d) Save all energy readings in an excel file as a record for lamp lifetime (date, type, desired current used, energy value reading before the test)

e) Once energy reading is stabilized, close window

2. Method Set-Up

- <u>New Method Set-up</u>

a) Go to File – New – Test Method – Choose element to test – Pick "Recommended Values" – Click OK

b) Method edition window will open.

c) In define Element,

i. Define method description (i.e. Cu02062015) – **Copy the name.**

ii. The spectrometer & signal values are automatically defined (i.e. wavelength, SR width...)

d) In Settings

I. Spectrometer: Set replicates value depending how many times the sample is to be retested. For manual, set delay time at 0. For Auto sampler, Set delay time at 20 (sec.)

II. Sampler:

i. in Flame, all values are fixed. No changes are needed

ii. In Auto sampler:

- Wash frequency – After all solutions
- Normal time -- 10 sec
- Wash location -- 0 (location of distilled water in Autosampler)

III. Calibration:

i. Equation and units : choose "Linear Calculated Intercept" and set all units to **mg/l**

ii. Standard Concentrations:

- In Calibration Blank, type BLK (usually water)
- In Reagent Blank, type the acid used to digest a sample
- In the Standard , do the following
  - ✓ Go to Tools – Recommended conditions
  - ✓ Pick element "Cu" and set Nebulizer to Stainless Steel
  - ✓ Look at the Sensitivity Check (for Cu, 4ppm for 0.2 abs.) Prepare ¼, ½, and x2 or max detection value specified for 4 ppm as standards.
  - ✓ In recommended conditions, check the needed oxidant (N2O or air). Acetylene is always used.
  - ✓ Check the remarks tab for anything additional that might need to be done.

- ✓ Close “Recommended Conditions” window
- ✓ In Standard Concentrations add the chosen names, concentrations & locations (in case of auto sampler)
- ✓ **Save Method (Very Important): Click Spectrometer tab -- copy name given – File - Save - Method-Paste name- Ok**

- Testing new samples Using an old method previously saved
  a) Click on Method tab – Choose the old method –Press OK
  b) When saving sample info and results data, sample info and results file names need to be changed

3. Flame Check
   a) Click “Flame” tab on top of screen to open Flame window
   b) Make sure a green check mark is shown
   c) For Air-Acetylene system, make sure Oxidant “Air” is selected
      I. C2H2 flow is 2.50 L/min
      II. Air flow is 10.00 L/min
   d) For Acetylene-Nitrous oxide system, make sure Oxidant “N2O” is selected
      I. C2H2 flow is 7.50 L/min
      II. N2O flow is 6.0 L/min
   e) Press ON to turn on the flame

4. Sample Information Set-up
   a) Click File – New – Sample Info File – OK
      I. Set Volume unit in (L) & Weight unit in (mg)
      II. Define ~~A/S location (in Autosampler if in use)~~, Sample ID, ~~initial weight & volume in case of digestion~~ ~~(all info are for the unknown sample(s) to check)~~
   b) Save Sample info (Very Important): File – Save as – Sample Info – Paste (same name given to method) – OK
   c) Close window
   d) To make changes to the File: Click Tools – Sample Info Editor -- make the changes, if needed.
   e) Once changes are made, click File- Save

5. Results Analysis Set-up (Manual Analysis)
    a) Click “Manual” tab on top of screen. Make sure flame is Idle (OFF).
    b) WE MUST SET IT UP TO SAVE THE RESULTS. **Save results (Very important):**
        i. Lamp window must be closed
        ii. Click “Open” under Results Data Set Name
        iii. In the name tack, right click - Paste (Paste the name given to method & sample info file) OR give a new name
        iv. Click OK
    c) Do NOT close window

6. Calibration Window
    a) Press “Calib” tab on top of screen to display calibration curve
    b) If old results are showing: Click Analysis – Click New Calibration

Perform Calibration (If needed)

a) Open Continuous Graphic Window by clicking on the tab on top of screen
b) Change max to 1 (for zooming)
c) Place test tube in water (the blank) and press “Auto Zero Graph” few times until it reads as close to zero
d) For Cu, test the 4 ppm standard. It should read 0.2 abs as per the “recommended conditions” window (under tools). If it does not read 0.2, do as follows:
    - Set the method to any Cu method in the system
    - Open “Continuous Graph” window
    - Zero graph with DI water
    - Aspire the 4 ppm Cu standard
    - Adjust nebulizer by turning the knob at end of test tube, if reading is not 0.2
        i. Clockwise SLOWLY to add
        ii. Counterclockwise to reduce
        iii. Close window when done
    - Adjust burner head (Remove tube from any samples)
        i. Turn off flame in Flame Control window
        ii. Click on aligned burner.
        iii. Choose “Automatically align the burner”. Click next
        iv. Turn OFF flame if it’s still on

v. Click Adjust. Burner will start to align
vi. Click OK if operation was successful
vii. Click Next.
viii. Turn flame back ON. Green checkmark box will appear
ix. Once flame is ON, place the solution that will give 0.2 abs. (4ppm) under the test tube
x. Click Adjust. Continuous graphics window will show a value that fluctuates until it stabilizes at around 0.2 for 4 ppm
xi. Close window then Press OK
xii. Click Finish in the Align burner window

e) Measure rest of samples. Make sure to dip test tube in water in between tests

7. Results Window
   a) Press "Results" tab on top of screen to display results
   b) If old results are showing: Click Analysis – Click Clear Results Display

8. Start the Test
   a) Turn ON flame to begin testing
   b) In the Manual Analysis Control Window, Press on:
      - Analyze blank (twice to reach a close to 0.0000 abs.)
      - Analyze standards
        ** To exclude a calibration point/ change calibration equation, click Tools -- Edit Calibration – Exclude point/change fit
      - Analyze Sample

9. To Recall/View old data
   a) Click "Tools" in Menu bar
   b) Click "reprint data"
   c) Click Open next to Results Data Set Name
   d) Highlight desired file to see. Click OK
   e) Click Open next to Analysis Batch date and Time
   f) Highlight the time of test. Click OK
   g) Press Preview. The results window will open.

10. To Reprocess data
    a) Click on “Reprocess” icon on top of screen (close manual analysis control window)
    b) Click Browse
    c) Choose method used
    d) Highlight in black the data to be reprocessed a press “Reprocess”
    e) One of the standards can be used as a sample to test. If value is same as tested before then we can proceed with new samples. If not, standards need to be prepared and a calibration curve created.
11. To Print or Export data
    a) Go to File – Utilities – Data manager
    b) Highlight the data result set in Blue
    c) Click on “Report” icon in Menu bar
    d) Click on “Next” on bottom of screen twice until we get to “Select Analytes To Report”
    e) Uncheck “All Analytes”
    f) Highlight in blue all data in the box
    g) Click on “Next”
    h) Click “Detailed” under report type
    i) Click “Preview”
    j) Click on the “Print” icon in Menu bar to print data or “Export” icon to export & save data file on desktop
    k) Click “Cancel” to exit

12. To flush burner head
    a) Aspirate Isopropanol for 10 min
    b) Aspirate 1% Nitric acid solution for 5 min
    c) Flush the drain tube thoroughly with water
    d) Empty the collection vessel and refill with water

13. To Shut Down
    a) Turn off flame
    b) Turn off lamp. Press on green circle
    c) Turn off software
    d) Turn off machine

e) Shut off compressor
f) Turn off hood #20
g) Turn gas valves off
h) Close gas tanks outside

14. To remove the Burner Head

To remove the burner head, press the tab on the safety latch found on the left side of the ignitor assembly with one hand, and gently pull and twist the burner head in an upward direction with the other hand until it comes off the chamber.

15. To install the Burner Head

Depending on the type of analysis you need to do, you may need to change the Burner head, see Burner Head Options on page 86.
1. Put the burner head back onto the burner chamber while pressing the safety latch tab.
2. Position the magnetic strip on the burner head ring around the back of the assembly - it should face the ignitor box (i.e. facing the right side). Release the safety latch. Be sure the burner head is fully seated on the chamber. A gentle twisting and pushing may be necessary. You will know that the burner head is fully seated because the ignitor will swing over the burner.

Link to a comprehensive manual:

https://www.manualslib.com/products/Perkinelmer-Pinaacle-900-Series-12081246.html