Perkin Elmer AA: Operating Procedure

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Prepare standards and samples before beginning this analysis.

1. Verify that the fume hood in ON. The hood extension over the AA is connected to the fume hood exhaust.

2. Turn on the AA 300 using the Power Switch on the lower right side.

3. Wait for the AA 300 to complete its startup sequence.

4. Log on to computer next to AA. Username = analab Password = Letmein!

5. Start AA Winlab Analyst from the desktop. After the software recognizes the AAnalyst 300, exit the test window.

6. Select Basic System Procedure, lower left green (flask) icon. Alternatively you can open this window by selecting: file – open – method.

7. The open method window is displayed. The default path is C:\AAUSER\METHODS, select method (5503 K Method). Click OK.

8. Click on the WkSpace icon in the tool bar. Select MANUAL.FLM, click OK. This displays the control windows.

9. In the Manual Analysis window click in the browse button next to the results data set name window. Enter a results name and description. Your results will be saved in the C:\AAUSER|RESULTS file under the name you create. Enter a sample ID.

10. Create a sample information file: Go to file – Save As – Sample Info File. Enter your file name using the .SIF extension.

11. Click on the “Standard 1” drop down menu in the manual analysis window. Notice that standards 1 through 5 have concentration values displayed below in the “Conc” window. This method is set-up for 5 standards of concentrations 1, 2.5, 5, 7.5 and 10 mg/L. If you want to edit these concentrations click on the MethEd icon in the top toolbar. Click on the Calib tab at the bottom of the window. Click on the Standard Concs tab on the right side of the window. Enter your concentration values for as many standards as you require. Enter zero for each corresponding A/S Loc window. Save the method in the default folder using a unique name. Close the method editor.

12. Other methods are located in C:\AAUSER\METHODS1. You may create your own method. Refer to the software manuals for complete information.

13. Select the Lamps icon from top tool bar, select “set up” for the lamp you are using. For analysis of potassium in juice select the (K, Na) lamp.
    The AA will automatically set up the selected lamp. This takes a few minutes. When the lamp setup is complete click (Set Midscale) – the bar graph should be at approximately mid scale, between 55 and 70. If the energy reading is below 50 consult lab personnel. Close the align lamps window.

14. THIS STEP IS REQUIRED ONLY IF THE AA HAS NOT BEEN USED FOR SOME TIME. Before lighting the flame, verify that the lamp is active and the burner is properly adjusted by placing a white card on the top of the burner perpendicular to the slot. Verify that the beam is centered over the burner slot. Do not look directly into the beam.

16. Open the main valve on the acetylene tank. Open the ball valve located on the wall behind the AA. The acetylene line pressure should read 13 psig. The air pressure gauge located on the AA gas purifier module located on the wall behind the AA should read between 60 – 70 psig,
    If the acetylene tank pressure is below 100 PSI do not light the burner, close the main valve and consult lab personnel. See page 1-15 of the hardware manual.

17. Select flame from the top tool bar or from the workspace window.
    Click on (Light burner). If a flame interlock error is displayed (No Drain) consult lab personnel. Under lab personnel supervision, remove the drain tube at the atomizer and pour about 150 ml of water down the tube. This will activate the sensor in the drain line. Once the checkmark in indicated in the safety interlocks window the burner should light. Click on the light burner icon. You may need to try multiple times to light the burner. If the burner does not light using the internal igniter use an external ignition source. Consult lab personnel for assistance. Once the burner is on, close the burner window.

18. Place the feed tube in a beaker of D.I water. From "Tools" select "Continuous Graphics", the instrument will run the lamp setup routine, select auto zero. Note the continuous graphics window is used to adjust the nebulizer. Adjustment of the nebulizer should not be required.

19. Close the continuous graphic window. Verify the feed tube is inserted in the D.I water. In the manual analysis window select analyze blank.

20. Wait until the indicator light in the analyze blank window is off then place the feed tube in the standard #1 container. Verify the drop down menu is set to display standard 1. Click on analyze standard.

21. After the standard has been analyzed place the feed tube in the D.I water for 30 seconds to flush out the line. Place the feed tube in standard #2 container, the Standard 2 should be displayed with the corresponding concentration. Click on analyze standard. Open the continuous graphics window to observe the real-time plot.

22. Repeat steps 17 and 18 until all standards have been analyzed.

23. Flush the line with D.I water as described in step 18. Place the feed tube in the sample container. Click on analyze sample.

24. Repeat as needed. Remember to flush the line between each reading.

25. Place the feed tube in a beaker of DI water. From “Tools” select “Continuous Graphics” select auto zero. Note, the continuous graphic window is also used to adjust the nebulizer. This procedure should have already been performed by lab personnel. See page 8-7 in the software guide for details.

26. Once the display has zeroed close the continuous graphics window.
Verify that the feed tube is in a beaker of D.I water. Select the manual analysis widow then select analyze blank.

27. Place the feed tube in standard #1. From the Manual Analysis window select analyze standard.

28. Once the indicator light in the analyze standard icon is off, repeat the analysis for each standard. Note the calibration curve developed from the standards. A report will print for the standards data.

29. Place the feed tube in the unknown sample. Select analyze sample. Repeat as needed.

30. Double click on the results window to show results details.

31. To print the calibration curve select the calibration window, from the file menu select print – graphics.

32. Close the continuous graphics window.

33. The data is displayed in the RESULTS window.
To print: A plot of the absorbance, close the results window then select PEAKS, CONTROLS, PRINT.
To Print: Results:
Select the results window, go to file – print –window image.
To print a report of the current data or data from a previous run, go to FILE – UTILITIES –REPORTER. Under DESIGN select CHRONOLOGICAL, COMPLETE, under DATA select the RESULT NAME of the file you wish to print. Select PRINT PREVIEW to view report. Select PRINT REPORT to print.

34. To Extinguish the Flame and Shut Down the System.

Rinse the nebulizer and burner system with suitable rinsing solutions, see below.
In the Flame Control window, click on the flame icon to turn off burner.
Turn off the burner gas supplies.
In the Flame control window, click on the Bleed Gases to depressurize the gas lines.
Wait at least 10 minutes after extinguishing the flame before turning off the vent system.
Exit the software
Switch off the spectrometer and log off computer.

Do take time to clean up.

Notes on cleanup:

Shutting Down the Analysis System after Flame Analyses

The procedure for rinsing the burner system depends on the type of samples that you have analyzed.

► If the sample solutions contained organic solvents, rinse all traces of the solvents out of the system. This is very important if you intend to analyze aqueous solutions next.

► If the sample solutions contained toxic substances, you must rinse all traces of these substances out of the system.

► If the sample solutions contained high concentrations of copper, silver or mercury salts, which can form unstable acetylides, you must rinse all traces of these substances out of the system.

Cleanup continued-----

With the flame still burning, aspirate the correct rinsing solutions in the sequence listed below to rinse the nebulizer and burner.

If you used only aqueous solutions during the analysis:

► Aspirate deionized water for five minutes.

If you used organic solvents during the analysis:

Aspirate for five minutes using an organic solvent that is miscible with both the solvent you used during the analysis and water.

Aspirate acetone for five minutes.

Aspirate 1% nitric acid for five minutes.

Aspirate deionized water for five minutes.

Either aspirate the solutions manually or use the autosampler.

Extinguish the flame and shut down the system.

Properly dispose of waste solutions.