

Atomic Absorption Spectroscopy

Atomic absorption spectroscopy (AAS) is a widely used technique for determining a large number of metals. In the most common implementation of AAS, a liquid sample containing the metal analyte is aspirated into an air-acetylene flame, causing evaporation of the solvent and vaporization of the free metal atoms. This process is called atomization. A line source (hollow cathode lamp) operating in the UV-visible spectral region is used to cause electronic excitation of the metal atoms, and the absorbance is measured with a conventional UV-visible dispersive spectrometer with photomultiplier detector. Figure 1 is a typical instrument block diagram. The light beam passes through the cloud of atomic vapor formed in the flame. The narrow spectral lines of gas-phase atomic samples necessitate the use of a line source as well as a high-resolution monochromator (i.e., a spectrometer capable of isolating a very narrow (e.g., < 1 nm) wavelength range). This helps to prevent interference from adjacent spectral lines of other atomic species present in the sample matrix. This allows a metal species to be analyzed directly in a complex sample without the need for performing separations. In this experiment, AAS in conjunction with flame atomization will be used to determine iron in the solution obtained through digestion of the unknown.

GBC Avanta Atomic Absorption Spectrometer Located in CTH 321



AAS Operation Procedure

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1. Make sure that computer is running the GBC software (double-click icon on Windows 95 desktop) and that the spectrometer is on (on/off button on side of AAS). Also, turn on the fuel and oxidant on the regulators on the tanks connected to the nebuliser (check both gauges on each tank).

2. Choose the Application Editor option to adjust settings for data collection:

Name—Choose a file name for your settings

Element—Type the abbreviation for the element in your solution

Matrix—The solvent you are using (probably water)

Wavelength—select an appropriate wavelength for your element and concentration range

Slit width—given for each element

Slit height—Normal setting

Measurement Mode—Integration

Calibration Parameters— Calibration Mode = Concentration

Flame Parameters— *Flame type* = Air or N₂O/Acetylene

Acetylene and Oxidant Flow = set ratios for reducing or oxidizing flame according to element (more air/N₂O = oxidizing, more fuel = reducing) *Burner Angle* = 0.0 degrees

Data Collection Parameters—*Replicates* = machine will take average of this number of measurements (5 is a common number)

Read Time = time in between replicate measurements (3 sec is common)

These parameters can be saved by hitting Enter at the Save Parameters option and can be recalled later by selecting Load Parameters on the Main Menu.

3. Open the lamp door on the left side of the spectrometer and turn the lamp holder to where the lamp labeled with the desired element is closest to you (the machine should prompt you to do this).

4. To perform a measurement run, choose the Run Parameters option from the Main Menu. Set the analysis mode at Random and the sampling mode at Manual (since this spectrometer is not equipped with a peripheral sample changing device).

5. Before you light the flame, ensure that the correct burner for your oxidant (air or N₂O) is in the nebuliser. To change the burner, pull the burner plug out of the right side of the burner compartment, turn the burner counter-clockwise and pull it out. Put the desired burner in place, turn it clockwise to lock into place, and plug into flow lines. Ensure that the burner is in the correct position by placing a white card over the burner slot. Use the Horizontal and Vertical adjustment knobs to adjust until the beam is parallel with and about 1 cm above the burner slot. The deuterium knob next to the cathode lamp can also be adjusted.

6. To ignite the flame, push the ignite button on the side of the AAS to light. Make sure that the flame sustains for about 20 seconds before continuing. The flame should be fairly small with little or no yellow color at the top and slightly pinkish at the bottom. The bright blue band at the bottom should be as thin as possible. To set the flame accordingly,

adjust the gas flow of the air, acetylene, and nitrogen tanks using the knobs on the tanks themselves, as well as adjusting the air/acetylene ratios on the computer under Flame Parameters.

7. Select the Alignment option on the Main Menu. This will display two dials that gauge the amount of signal reaching the spectrometer. With one of your samples aspirating (a moderate concentration) (see #8- instructions for aspirating) adjust the Vertical alignment to where the dial on the left is approximately in the middle of the gauge while not decreasing the reading on the dial on the right. This is to ensure that extremely high/low concentrations do not read absorbances that are too close to the maximum/minimum sensitivity of the spectrometer. (See Minimum Measurable Absorbance section of discussion). Also ensure that this does not disrupt previous alignment performed in step 5.

8. When you are ready to begin the calibration procedure, hit F2 to go to the results screen. Hit F10 to take data on a sample. The machine will ask for the sample in a new dialog box. At the beginning of your runs, you should test a water sample as a blank. Select the Solution as Cal Blank. On the nebuliser, make sure that the apparatus is aspirating by watching for fluid coming out of the trap into the waste tube (or by aspirating a concentrated sodium solution.

The flame will turn bright yellow). Place the capillary tube in DI water (idle machine should already be aspirating water). Select Read on the dialog box. The AAS will collect the specified number of measurements and average them. This will zero the machine.

9. Aspirate each calibration standard in the same manner, only choosing Sample as Solution. Wipe off the capillary tube with a Kim-wipe and place it back in DI water between each standard.

10. After you have taken data on all standards, plot the absorbance vs. concentration on Excel or other data program. If the machine is working properly, the data should be relatively linear. (For sample plots, see Appendix 3.) If the data is not linear, then certain parameters will have to be varied to make data more linear for the determination of unknown concentrations with Beer's Law (see discussion).

11. To turn the machine off, extinguish the flame by pressing the ignite button on the AAS. Turn off the pressure on regulators on the fuel and oxidant tanks. Then select the Bleed Lines option from the Flame Parameters option in the Applications Editor. This will remove any excess gas from the system. Exit the GBC software by hitting Escape (ESC) until the system goes back to Windows.

Common Problems and Fix-it Strategies

Problems with the instrument

1. Many different problems are possible if the flame will not light or will not remain lit. In order to diagnose these problems, go to Status under Flame Parameters in the Applications Editor and attempt to ignite the flame. Under Last Shutdown, the reason for shutdown will be given. Some of the more common ones were:

(a) **Low level in trap**— need to refill the liquid trap with whatever solvent you are using. Unscrew the white cap on the top of the trap and use a funnel to pour solvent until you see it leaking out of the waste tube. It also necessary to periodically make sure that the waste tube is not backed up and empty it into the waste sink.

(b) *Insufficient fuel/oxidant or low pressure* — make sure that regulators show flow of both fuel and correct oxidant. Check for leaks in fuel line and regulator connections using soapy water.

(c) *Burner is not connected*—after switching burners the burner plug may not have been plugged in, doing this will solve the problem. Additionally, the air current in the hood is sometimes strong enough that it interferes with the ignition flame itself, pulling it away from the fuel flow out of the burner. To minimize this, cover the opening with a piece of paper while igniting.

2. It is also important to make sure that the aspiration rate is sufficiently high to get sample into the nebuliser. Under normal conditions, the capillary tube should evacuate almost immediately when removed from solution or water. If this is not the case, there are a few steps that you can take:

(a) Make sure that the aspirator knob is turned sufficiently far to the left (too far to either side will not allow proper aspiration).

(b) Make sure that the capillary is clean and not clogged with solid deposits.

(c) As a “last resort”, you can manually evacuate the tube in between each sample by pulling the tube off the nebuliser and holding the capillary tube to the house vacuum in the vent hood, creating a seal around the outside with your fingers.

3. Soot and corrosion can also collect on the slit in the burner, creating a non-uniform flame that can interfere with measurements. Periodically, the burner slit needs to be cleared by sliding a small card back and forth through the slit WITH THE

FUEL/OXIDANT FLOWS

OFF.

4. If after taking a sample absorbance the absorbance value is very low (<0.010) or entirely out of range with expected values, the error message “HC low lamp intensity” may be given. It is likely that the burner is improperly aligned assuming all other parameters are correct (may need to check aspiration rate). Refer to steps 5 and 7 of the Procedure for instructions. It is also possible that the lamp has burned out and needs replacing. (Lamps on average last about 2 years.)

Problems with the computer program

1. A common error message, “Absorbance not increasing with concentration,” is often given even when the absorbance is in fact increasing. The program will prompt you to hit the space bar to clear. If you do this and attempt to run another standard or sample, the machine will take your standard/sample and zero it. The best option is to ignore the message and continue running your standards/sample by hitting F10.

2. It is easier to keep track of calibration tables and parameters by creating a new method each time you run a different standard curve or standard addition on the machine. The program has a tendency to default to the first calibration tables and standard readings performed, as well as requiring you to recalibrate any time your standard run is interrupted or you want to run a new set of standards. Simply type a new name after “name” in the Application Parameters menu and save. Remember to name and save your calibration table as well.

Reagents & Solutions

1. Atomic absorption standard solutions of CuCl_2 is available in CTH321.

2. Prepare 50 ml of 100.0 ppm Cu stock solutions from a 1000 ppm stock available in the lab. (Please do not pipette from the reagent bottle – pour an appropriate amount into a beaker for this)
3. From your stock solution prepare 100 ml of a 1.00 ppm Cu standard solution and 50.0 ml of the following Cu standard solutions: 0.150, 0.200, 0.250, 0.500 ppm Cu.