Atomic Absorption Spectrometry

In atomic absorption spectrometry (AA) the elements present in a sample are converted to gas phase atoms in the ground state.The UV-Vis absorption of these gas phase atoms are then measured by irradiation of light at a highly specific wavelength causing a transition of some of the gas phase atoms to a higher energy level.The extent to which light is absorbed is related to the original concentration of ground state atoms.This situation is completely analogous to the Beer-Lambert law in conventional liquid UV-Vis absorption spectrophotometry.

The most common instrument components consist of a hollow cathode lamp source, a pneumatic nebulizer for an atomizer, a conventional grating monochromator and photomultiplier tube detector.The hollow cathode lamp is made of a glass envelope with a quartz window filled with an inert gas at slightly above atmospheric pressure.The cathode is made of the pure metal of interest (Cr if you are doing a Cr analysis, Pb for a Pb analysis, etc.).The pneumatic nebulizer aspirates and nebulizes the liquid sample solution when the sample is sucked through a capillary tube.The grating monochromator eliminates much of the background light from the flame and the photomultiplier tube detector detects that light from the hollow cathode lamp which passes through the flame.

Atomic absorption analyses are most commonly and routinely performed on solutions.Therefore your sample must be converted to liquid form prior to analysis.This is most conveniently done using a microwave to digest the sample, leaving a solution that can then be analyzed.(*Note that the procedure outlined to digest your soil uses concentrated HNO_3.This does not compleletely dissolve the soil, as SiO_2 is insoluble in concentrated HNO_3.Thus Prior to AA analysis you must filter the solution as particulates can clog the nebulizer.This problem could be circumvented by the use of HF, but given the extreme health hazards associated with HF it will not be used.)*There must be a sufficient concentration of analyte for the spectrometer to detect.Prior to using the AA for your analysis, you will need to determine the minimum detection limit for the element of interest.The minimum quantifiable limit (the lowest concentration of analyte

which can be quantitatively determined) is generally 3-5 times the minimum detection limit.

The minimum detection limit is intimately related to the concept of signal-to-noise ratio (S/N) and the standard deviation of a measurement.Noise is conveniently measured as the standard deviation of numerous measurements of the signal.It is possible to detect a signal when the S/N is three or greater.Thus the minimum detection limit is when the S/N = 3.If you measure the standard deviation of a number of measurements you can then calculate the minimum detectable signal.The minimum detection limit is obtained by experimentally determining the concentration of analyte which gives the minimum detectable signal.From the minimum detection limit calculate the minimum quantitiable limit.Check your calculations to see if the AA will detect the analyte concentration you expect in your sample.If you cannot detect that low of a concentration, decide if a preconcentration step is in order.

General

- A well established technique for the quantification of nearly 70 elements in a variety of sample types with sensitivity at the ppm level or less. Aqueous samples can be determined with no sample preparation, solid samples must be dissolved or digested. This sample preparation for solids can be time consuming.

Common Specific Applications

- Wherever quantitative elemental analysis is important: biological, medical, clinical, environmental, pharmaceutical, foods, etc.

Limitations

- Elemental range is limited to metals and metalloids.
- Sample preparation is tedious and time consuming.
- The sample is destroyed by the analysis.
- Only one element at a time can be measured.

Complementary or Related Techniques

- Inductively Couple Plasma (ICP) atomic emission spectrometry provides parts-per-billion sensitivity and can perform multielement analysis. Supplanting AA in many applications.
- XRF* can perform multielement analysis and is particularly well suited for

solid samples. Quantitative analysis is possible but a bit tricky due to matrix effects.

- Flame Emission Spectrophotometry* is only usable for a handful of elements which can be excited in a relatively low energy flame. Flame emission requires a dissolved sample.
- Neutron Activation Analysis has high sensitivity but requires a nuclear reactor for activation. It is the ultimate in elemental analysis in many respects, but is generally useful for only specialized applications.

* - Available to students in instrumental analysis at EIU.

References used to devise this web page:

1. "Handbook of Instrumental Techniques for Analytical Chemistry"Frank Settle, Editor:"X-Ray Fluorescence Spectrometry", G.J. Havrilla.Prentice Hall 1997.
2. "Principles of Instrumental Analysis", 5th Edition by Skoog/Holler/Nieman, Saunders College Publishing 1998.

Additional References

1. "Spectrochemical Analysis" Ingle and Crouch, Prentice Hall, 1988.
2. "Spectrochemical Analysis by Atomic Absorption and Emission, L.H.J. Lajunen, Royal Society of Chemistry, 1992.

A few decent web sites on AA

1. A good overall web site for a variety of information on spectrochemical analysis:Spectroscopy home page