

Atomic Absorption Spectroscopy (AAS) Analysis

Before starting your first AAS analysis, you should watch the video tape provided by Professor Stucki. It gives a detailed demonstration of the proper use, procedures, and settings for the Varian Model SpectrAA210 spectrometer for AAS analysis.

Preparations

Prior to AAS analysis, preparation of sample solutions is required, followed proper protocols depending on the type of analysis being performed (absorption, emission, element(s), etc.).

The Varian instruction booklet entitled "*Analytical Methods*" lists all the chemical elements one can identify with AAS. It is recommended to go through this book and consider the element one wants to analyze.

Table 1 provides some indications concerning two elements commonly analyzed in Professor Stucki's group.

Table 1: Sample preparation for AAS analysis		
Element	Sample preparation	Interferences
Na	To analyze Na it is recommended to use Cs ⁺ as an ionization suppressor. A final concentration of 0.1% (1mgCs ⁺ /ml) is a suitable value and is obtained by adding CsCl to the samples as well as to the standards.	Na is an element which is present in numerous stuff, e.g. towels, skin... Glassware too frees a lot of Na. Hence it is recommended to be very careful with Na analysis and follow this advice: -prepare solutions in plastic containers, do not use glass. -wear disposable gloves, a clean lab-coat...
Fe	If the samples contain Fe in a precipitated form, dissolution is required. Add HCl to the solutions in order to obtain a pH<2. The acidified solutions have to remain 3 days before analysis.	(See Loveland (1988) for more information about Fe analysis)

Preparation of Standards

The establishment of a calibration curve is obtained by standards which have to be prepared in the same way as the samples. Thus, the standards must also be treated as described in Table 1.

Starting the AAS Analysis

- Turn on the Varian software and the spectrometer
- Align the burner
- Prepare the software settings (The video tape contains detailed information about the software programming)
- The light has to be switched on at least 20 minutes before the analysis, in order to let the light beam stabilize
- Open the valve of the hood above the spectrometer
- The acetylene flame has to be turned on some 10 minutes prior to the analysis, to heat the burner up to a constant temperature
- Use fresh distilled water and always leave the sucking tube in it
- Measure the sucking flow by using a chronometer and a graduated cylinder. A flow rate of 5 ml / minute is an accurate value. The flow rate can be modified by adapting the position of the black screw which fastens the sucking tube.

Procedure of AAS Analysis

- Between the readings of any two samples, the sipper tube must remain in a solution. It is recommended to use the background matrix of your samples as a rinsing solution, e.g., if you want to analyze Na in a MgCl_2 matrix, you should rinse the system with a blank MgCl_2 solution of the same molarity.
- Start by setting the calibration curve with at least 5 standards. The concentration range depends on the specific element. Refer to the Varian instruction

booklet entitled "*Analytical Methods*" for the linear range of the absorbance vs. concentration relationship.

- After the last standard, a long rinsing time of 1 minute at least is required, as the last standard is the most concentrated and precedes immediately your first sample.
- Between each analyzed solution a rinsing time of 30 seconds with the rinsing solution is suggested, to clear out all influences from the previous sample. After sipping the sample solution one should wait for 10 seconds before requesting the software to read the absorbance. This allows the system to be filled up with the sample solution.
- After 10 samples it is recommended to analyze one of the standards used to establish the calibration curve. The comparison of the obtained absorbances gives an indication about the stability of the analysis.
- Between samples, the reading of a "spy sample" allows you to get an idea of the accuracy of your analysis. A spy sample is a sample which is prepared from a certified reference solution in the same way as the standards. However, it should be prepared independently from your standards, which means that it should not derive from a standard. The discrepancy between the expected concentration and the measured concentration (according to the AAS absorbance) of the spy sample is a good way to evaluate your results.
- At the end of the analysis the sipping tube should remain for 5 minutes in fresh distilled water, to rinse the system completely.

After the AAS analysis

- Turn off the flame, the spectrometer and the software
- Close the valve of the fume hood
- Let the burner cool down
- Immerse the burner in nitric acid (HNO_3 5 % v/v) for 12 hours
- Rinse the burner abundantly with distilled water and let it dry before installing it in the spectrometer

Literature Cited

Loveland, Peter J. 1988. Assay for iron in soils and clay minerals. Pp. 99-140 (Chapter 6) In *Iron in soils and clay minerals*, Stucki, Joseph William, Bernard A. Goodman, and Udo Schwertmann (Eds.). D. Reidel, Dordrecht.

-- Christian Bogdal, December, 2004