



Measurements with Atomic Absorption Spectrometer (AAS)

Introduction

The atomic absorption spectrometry (AAS) is a selective analytical method based on the absorption of radiation by free atoms in the ground state. A given wavelength of an element corresponds to an optical transition between atoms in the ground state and excited level. The absorption is related to the concentration of the atoms and thus to the concentration of the element. By measuring the intensity of the absorbed radiation the amount of an element in the sample may be determined.

Principle

The principle of detection is governed by Beer's law which says that the transmitted radiant power will be diminished exponentially with increasing absorption coefficient as well as with increasing length of the absorbing layer. A continuum radiation source passes through the absorption cell filled with the atomic gas of the sample having an absorption line with a characteristic wavelength. The absorption coefficient will be a function of the wavelength.

Equipment

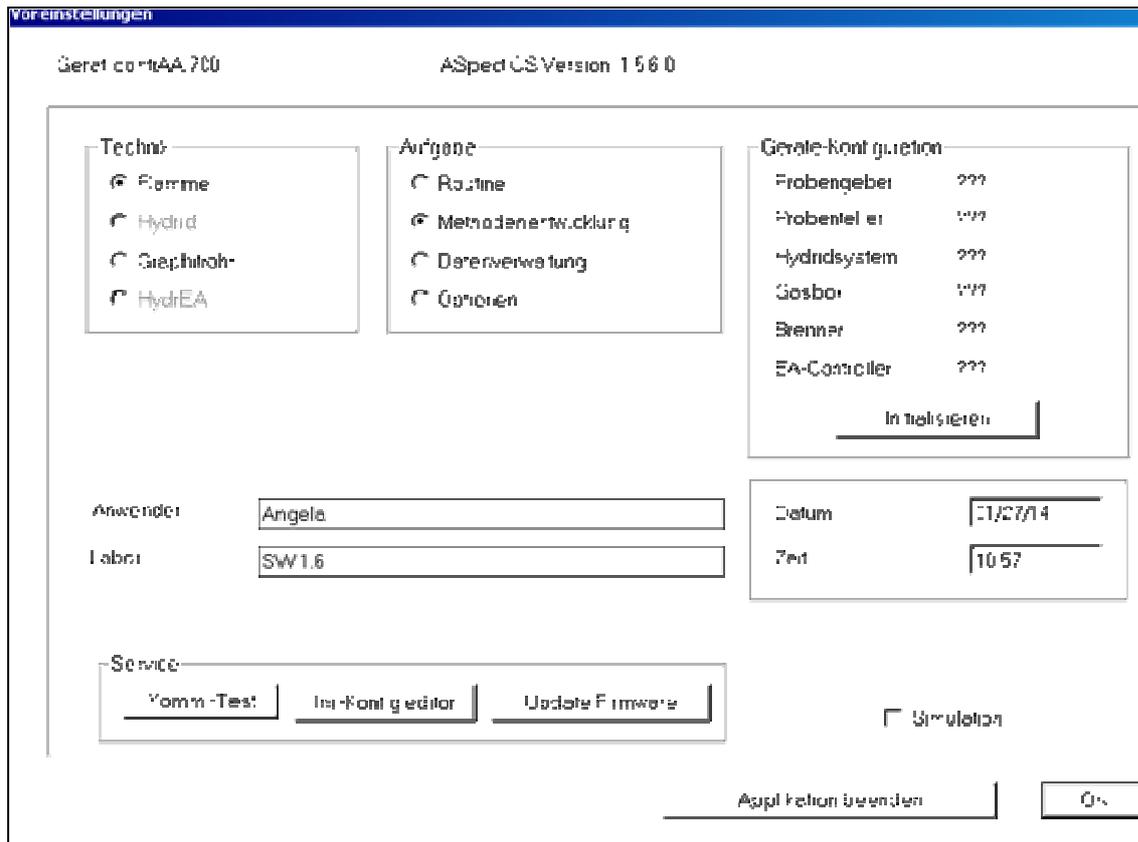
In the BfS a high resolution continuum source AAS instrument is used for atomic absorption measurements (HR-CS AAS)

(Fig. 1) Fig. 1 High-Resolution Continuum Source AAS



As continuous source a high-pressure xenon short-arc lamp is built in. Two types of atomizers can be used: the flame or the graphite furnace. The flame atomizer with acetylene-nitrous oxide system has a temperature of around 2700 °C and can be automatically cleaned after each measurement. The graphite furnace consisting of a transversely heated graphite tube with platform assures a homogeneous temperature distribution during measurements and shorter heating-up periods. Generally the furnace provides lower detection limits but the measurement takes much longer. These issues should be considered before choosing the atomizer (Fig. 2).

Fig. 2 Choice of the atomizer (here flame is selected)



As detector a very sensitive CCD semiconductor is used, which is also employed in ICP-OES. This allows the detection in the wavelength range between 185 nm and 900 nm making possible simultaneous measurements over a wide spectral range. The instrument has a sample changing device with 89 positions and can automatically identify too high concentrations and accordingly adjust the dilution of the samples. The solution may be diluted by a factor of up to 10. Within the graphite furnace a further dilution by a factor of up to 6000 may be applied. Furthermore, it provides automatic or manual background correction. It contains an automatic baseline adaptation. The operation, control and analysis can be done with the same software ASpect CS ver. 1.5.6. There are fully automatic and manual modes available. Depending on the concentration in the sample, each analytical line of the respective element can be used for analysis.

Technique

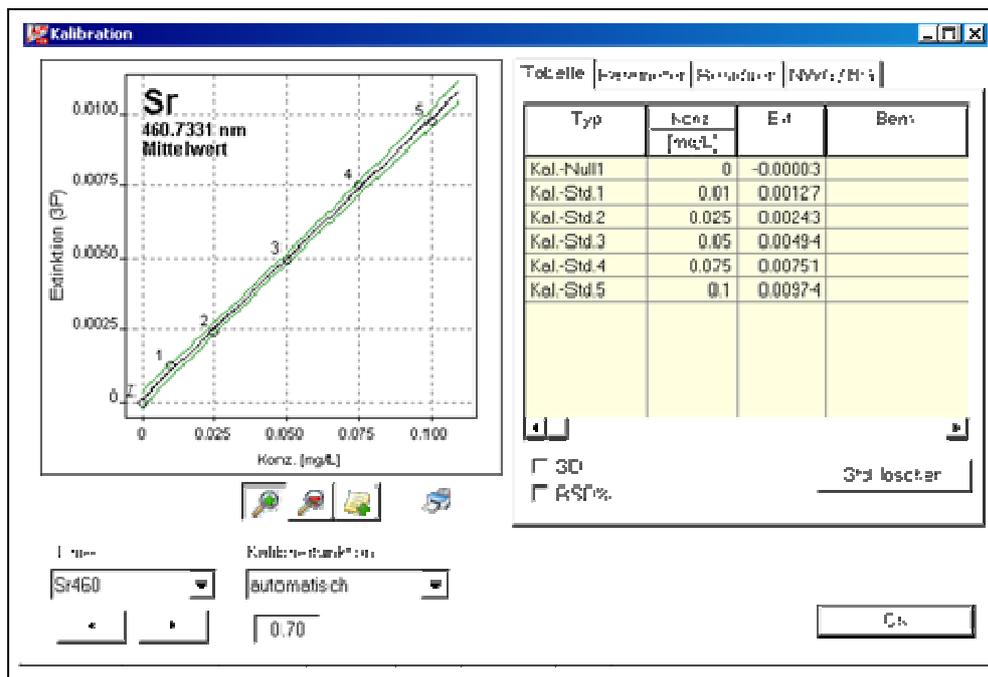
In case of the flame technique, before starting the sample measurement the background is measured using (bi-) distilled water. Then a reagent blank is measured. For the calibration curve a series of standard solutions with increasing concentration are prepared from a certified AAS-standard and measured. The concentration range of the series should be within the range where the concentration in the sample is expected.

Before the samples are measured, the adjustment of the instrument has to be carefully done. This involves the setup of the method parameters and sequences. The sample supply can be done manually after optimisation or semi-automatically.

If the graphite furnace is applied, the temperature programme should be preset and optimised as well by means of an internal camera. Furthermore, the camera helps to monitor the quality of the atomization process. For determinations with the graphite furnace only one standard (~ 500 µg/l) is used. The sample supply is done through the sample changing device. For each element a separate procedure is used which has to be newly created or selected from the existing list.

A typical calibration curve for strontium analysis can be seen in Fig. 3

Fig. 3 Generated calibration curve with 5 standards with increasing concentration



Using the calibration data and the blank corrected absorbance values for each sample determined after 3 to 5 repeated measurements, the analyte concentration is calculated for each sample.

Applications

The BfS is using two identical instruments at two different locations. The technique is applied e.g.:

- for the determination of stable chemical elements in order to calculate the chemical yield during and after a radiochemical analysis;
- to identify disturbing elements in a sample matrix;
- to determine the total loss factor in air filter samples from NPPs;
- to quantify the chemical yield for water samples from NPPs.

The achieved detection limits calculated according to the calibration curve method are summarised in the Table 1.

Table 1 Detection and quantification limits with the HR-CS AAS with different atomizer techniques

Flame technique (acetylene/nitrous oxide) (white)		Graphite furnace technique (Transversely heated graphite tube with platform) (in gray)	
Element	Detection limit	Quantification Limit	Standard solutions used
Mo	70 µg/L	250 µg/L	0,1 mg/L – 1,0 mg/L
Mo	1 mg/L	4 mg/L	5,0 mg/L – 20,0 mg/L
Mo	1 µg/L	4 µg/L	10,0 µg/L – 50,0 µg/L
Pb	ppm	ppm	
Re	1 mg/L	2 mg/L	1,0 mg/L – 10,0 mg/L
Re	21 µg/L	83 µg/L	125,0 µg/L – 250 µg/L
Sr	4 µg/L	15 µg/L	0,01 mg/L – 0,1 mg/L
Sr	400 µg/L	2 mg/L	1,0 mg/L – 10,0 mg/L
Sr	1 µg/L	4 µg/L	5,0 µg/L – 40 µg/L
Ti	16 mg/L	56 mg/L	60,0 mg/L – 175,0 mg/L
Ti	4 µg/L	15 µg/L	10,0 µg/L – 75 µg/L
Y	2 mg/L	5 mg/L	1,0 mg/L – 10,0 mg/L