Application Notice #AA-14

The Determination of Silicon by Flame AAS

Introduction:

Flame AAS with a nitrous oxide/acetylene flame is often a favorable method for the determination of silicon because of its ease of use and high sensitivity. However, the sensitivity and measurement precision very quite drastically depending on the spectrometer used as well as the flame and chemical conditions. This application notice summarizes the parameters that are critical to the optimization of flame AAS for the analysis of silicon.

Important Factors to be Considered:

1. The Flame and its Stoichiometry:

The sensitivity for silicon depends significantly upon the stoichiometry of the nitrous oxide/acetylene flame. The best sensitivity is obtained with a fuel-rich flame. For example, using nitrous oxide and acetylene flow rates of 11 and 5.0 L/min. respectively, the absorbance of a 100 ppm silicon solution was about 0.5. However, if the acetylene flow rate was reduced to 4.0 L/min. the absorbance of a 1000 ppm silicon solution might not be detected. The operator must carefully optimize the fuel rates of his or her system to ensure maximum performance. The data supplied by the manufacturer can be used as a reference, however, because many commercially available instruments (other than the AI 1100) rely on a gas flow calibration the values displayed by the instrument may not be the actual flow rates.

2. Bandwidth:

The most sensitive line for silicon is at 251.6 nm, however, the silicon spectrum around this line is quite complicated, as can be seen in figure 1. As a result, a narrow bandwidth is essential to resolve this line and thus achieve the best possible sensitivity and most linear calibration curve. It has been proven with experiments that the use of a bandwidth higher than 0.7 nm will significantly decrease the sensitivity and degrade the linearity of the calibration curve. The optimum signal-to-noise ratio and an acceptable linearity can be obtained using a bandwidth of 0.2 nm. Slightly further improvement in linearity is observed with a smaller bandwidth, however, the increased PMT voltage which is required results in poorer signal-to-noise ratios.

Figure 1: Wavelength scan of a Si HCL around the 251.6 nm line.

3. The Acidity of the Sample:

The determination of silicon in a nitrous oxide/acetylene flame is virtually free of interferences, however, the fact that silicon is rapidly preciptitated from acid solutions can cause difficulties. It is recommended that any silicon solutions be prepared in basic media.

In addition, the burner height and lamp current should also be optimized. It is easiest to use a relatively highly concentrated solution at the beginning of the optimization to ensure a signal can be seen.

Typical Instrument Conditions:

Below are some typical conditions for silicon determination with a nitrous oxide/acetylene flame using Aurora Instruments' AI 1100 Atomic Absorption Spectrometer:

Parameter	Suggested Setting
Bandwidth	0.2 nm
Lamp Current	12 mA
Wavelength	251.6 nm
PMT voltage	336 V
Integration time	5 seconds
Acetylene flow rate	5.0 L/min.
Nitrous oxide flow rate	preset

Results:

Figure 2 shows a typical calibration curve obtained under the conditions described above. The characteristic concentration was calculated to be 0.9 mg/ml. The RSD of the measurement was found to be less than 1%.

Figure 2: Calibration curve for Si by nitrous oxide/acetylene flame

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