# Application Note

## Sensitivity Improvements in Flame AAS Using the GBC Atom Trap

### Introduction

Standard flame atomic absorption spectrometry (AAS) is a well established technique with well defined analytical parameters for 68 elements. In recent years small improvements in nebulizer, spray chamber, and burner design have occurred, but the total effect on the atomization process has been small and other analytical techniques have been employed to improve sensitivity. These include concentration procedures such as:

1. Evaporation. This has the disadvantage of concentrating the sample matrix as well as the analyte.

2. Solvent extraction procedures. These result in a clean sample, however the sample preparation is time-consuming and may not be suitable for some types of samples, and

3. Ion exchange separation of metal ions. This is useful only for samples in a simple water-based matrix. In using these procedures the potential for contamination cannot be ignored, hence the interpretation of results may be difficult.

Hydride generation has been utilized to dramatically improve sensitivity and provide a clean sample at a low concentration in the μg/L (ppb) range. However it is available only for the hydride forming elements and mercury. Graphite furnace atomization is a major step toward sensitivity improvements (up to 1000 times) however it requires specialist knowledge of the technique and a major investment.

The use of a slotted quartz tube to increase sensitivity in flame AAS was first described by Watling in 1978[1]. This procedure required the slotted tube, held in a metal bracket, to be positioned above the burner slot in the air-acetylene flame such that the light beam from the hollow cathode lamp passed through the quartz tube. The tube had two slots cut to allow the flame to enter and leave the tube. The flame entered via the longer slot, exited through the shorter slot. The effect was to retain the atoms in the tube and increase the atom density in the lightpath. The result was a dramatic improvement in sensitivity for particular elements. (See Figure)

This document reports on the improved sensitivity for flame atomic absorption using the air-acetylene burner in combination with the GBC Atom Trap.

### Experimental

#### Instrumentation

A GBC 906 double beam atomic absorption spectrophotometer (AAS) equipped with ultra-pulse deuterium arc background correction system was used. The GBC Atom Trap was installed on an air-acetylene burner. A GBC data station (AT-compatible computer with 40 megabyte hard disk drive), with 906 AAS operating software installed, was utilised for developing the flame atomic absorption applications used for collecting and storing data, and displaying all graphics traces. The real time colour graphics traces for the standards and sample signals allowed the analytical conditions to be optimized. Method parameters, results, and graphics traces were printed for each analysis.

For each element, recommended GBC flame analysis conditions for wavelength and slit width were used[2]. Super Lamps were used for cadmium, lead, selenium and tellurium. Standard hollow cathode lamps were used for all other elements. The recommended operating current for each lamp was used.

The GBC Atom Trap cell (see Figure 1) is made of high grade quartz (Heralux ™). The slots are produced by diamond cutting. The mounting bracket is made of corrosion resistant steel with twin mounting spring clips, and is placed on the burner and clamped in position. It should be noted that the use of nitrous oxide-acetylene is not practical since the higher flame temperature produced, approximately 2,600 °C, would damage the quartz tube.

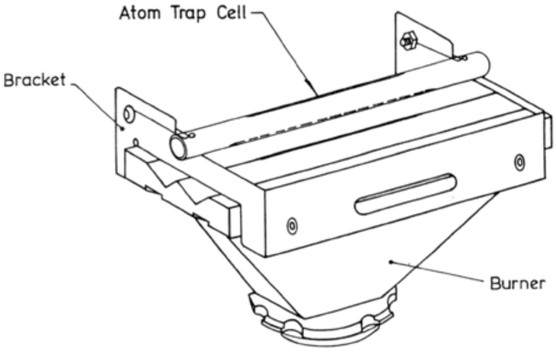

***Figure 1:*** *Diagram of the atom trap cell clamped onto its support bracket mounted on an air-acetylene burner.*

## Reagents and Sample Preparation

All chemicals were analytical grade. Nitric acid ($HNO_3$) Aristar-grade, was obtained from BDH Chemicals (Australia). Atomic Absorption Standards for each element of 1000 μg/mL were also from BDH Chemicals (Australia). Deionized water for washing and rinsing was obtained from a mixed-bed deionizing unit (Service Exchange Deionization System, Continental Water Systems Pty Ltd). Deionized water used for reagent preparation and analysis was from a reverse osmosis, mixed-bed deionizing unit that supplies Type 1 ultrapure water (Modulab, Reagent Grade Model Water Systems, Continental Water Systems Pty Ltd). Analytical samples were freshly prepared each day at the appropriate concentration for each element. Background correction was used for all elements except copper and iron. Solution blanks and samples were acidified with nitric acid to give 1% v/v in final volume. To determine the characteristic concentration for each element, 10 replicates of 10 seconds integration time were run, interspersed with blank.

Sample concentrations were chosen at approximately ten times the detection limit (silver 0.02 μg/mL, gold 0.01 μg/mL, bismuth 0.4 μg/mL, cadmium 0.004 μg/mL, copper 0.01 μg/mL, iron 0.05 μg/mL, mercury 1.5 μg/mL, manganese 0.015 μg/mL, lead 0.1 μg/mL, antimony 0.4 μg/mL, selenium 5 μg/mL, tellurium 0.2 μg/mL and zinc at 0.005 μg/mL). For comparative purposes, measurements were made without the atom trap and then repeated with it in place. The only change in the application parameters was the setting for burner height which needed to be adjusted appropriately for each element.

## Characteristic Concentration

Characteristic Concentration of an element has been defined as its concentration in solution which produces a change, compared with pure solvent, of 0.0044 absorbance units (1% absorption) at the wavelength of the absorption line employed.

$$\text{Characteristic Concentration} = \frac{0.0044 \text{ x concentration}}{\text{mean absorbance}}$$

**Characteristic Concentrations (μg/mL)**

| Element | Standard Flame | Atom Trap | Improvement Factor |
|---|---|---|---|
| Silver | 0.015 | 0.0075 | 2 |
| Gold | 0.05 | 0.03 | 1.6 |
| Bismuth | 0.100 | 0.035 | 2.9 |
| Cadmium | 0.0086 | 0.0025 | 3.4 |
| Copper | 0.022 | 0.009 | 2.4 |
| Iron | 0.030 | 0.019 | 1.6 |
| Mercury | 1.3 | 0.42 | 3.1 |
| Manganese | 0.016 | 0.005 | 3.2 |
| Lead | 0.060 | 0.0215 | 2.9 |
| Selenium | 0.20 | 0.06 | 3.3 |
| Antimony | 0.19 | 0.06 | 3.1 |
| Tellurium | 0.25 | 0.07 | 3.6 |
| Zinc | 0.005 | 0.003 | 1.6 |

***Table 1:*** *Comparison of Characteristic Concentrations for a range of elements, determined with/out the GBC Atom Trap.*

## Results and Discussion

All elements determined showed an improvement in Characteristic Concentration when using the atom trap with the majority showing 2 to 4 times improvement (See Table).

As a result of this increase in sensitivity for these elements, the operator can measure concentration values previously believed to be non-detectable by standard flame AAS. The advantages of this device are in the analysis of environmental samples, and particularly water quality testing, for elements at low concentrations which may not have been measurable by standard flame analysis. The operation of this accessory is simple and fast, as the holder is attached to a standard air-acetylene burner and the quartz atom trap clamped in the light path. Standard flame parameters are used for each application with the only adjustment required being optimization of burner height. In routine analysis the use of the atom trap is almost identical to standard flame analysis but the improvements in sensitivity are dramatic. In terms of speed of operation, once the accessory is installed, analysis time is as fast as flame analysis and as convenient.

## References

1. Watling, R.J., Anal. Chim. Acta 97, 395 (1978)
2. GBC Flame Methods Manual for Atomic Absorption