FIELD APPLICATION REPORT

Atomic Absorption

# Determination of Tin in Bead-wire Coating by Flow Injection Hydride Atomic Absorption Spectroscopy

## Introduction

The tin (Sn) content of bead-wire coating can be determined using flow injection (FI) analysis with hydride-generation atomic absorption (AA) spectroscopy, following recommended conditions.[1] These recommendations for Sn analysis include extensive conditioning of the quartz cell in 40% concentrated hydrofluoric acid (HF) to maintain optimum sensitivity. A modified system design is described here in which a small amount of air is mixed with the argon, which improves the signal dramatically and eliminates the need for HF cell conditioning.

## Experimental

### Sample Preparation

The tin wire was wiped gently with a soft cloth moistened with petroleum ether and then cut into 3-cm segments. The filaments were rinsed with petroleum ether, then acetone, and then dried in an oven for 15 minutes at 105 °C. They were cooled in a desiccator and then approximately one gram of sample was selected and weighed to the nearest 0.1 mg. The brass plating was dissolved by covering the wire with 15 mL of a 50% ammonium hydroxide solution. Then 0.5 mL of 30% hydrogen peroxide was added. Immediately after the plating had dissolved (usually a minute or less), the solution was decanted quantitatively into a 100 mL beaker. The wire was rinsed with water and the water was added to the solution in the 100 mL beaker. The stripped wire was rinsed with acetone and dried in an oven for 15-30 minutes at 105 °C. It was then cooled in a desiccator and weighed to the nearest 0.1 mg to determine the weight of dissolved plating.

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The decanted solution in the 100 mL beaker was boiled until the excess hydrogen peroxide and the ammonia were driven off. The solution turned dark-cloudy due to the precipitation of the copper oxide. The solution was then cooled to room temperature, acidified with 1 mL of concentrated HCl and transferred quantitatively to a 100 mL volumetric flask. The solution was made up to the 100 mL mark with water. A blank was prepared in an identical manner, with the omission of the sample. The sample and blank were each diluted (1:10) with $H_3BO_3$ solution (approximately 5% w/v in 1% v/v HCl).

## Instrumental

A PerkinElmer® FIAS-400 flow injection system was used for the analysis and a modified tubing arrangement was employed (Figure 1). The outlet from the gas/liquid separator to the spectrometer was connected via a type III T-connector (PerkinElmer Part No. B0199035). A 1/8" tube was connected to the T-piece after winding through Pump 2 to allow some air to be pumped into the system. This allowed argon gas carrying sample to be mixed with some amount of air (oxygen). The FIAS-400 program used is shown in Table 1.

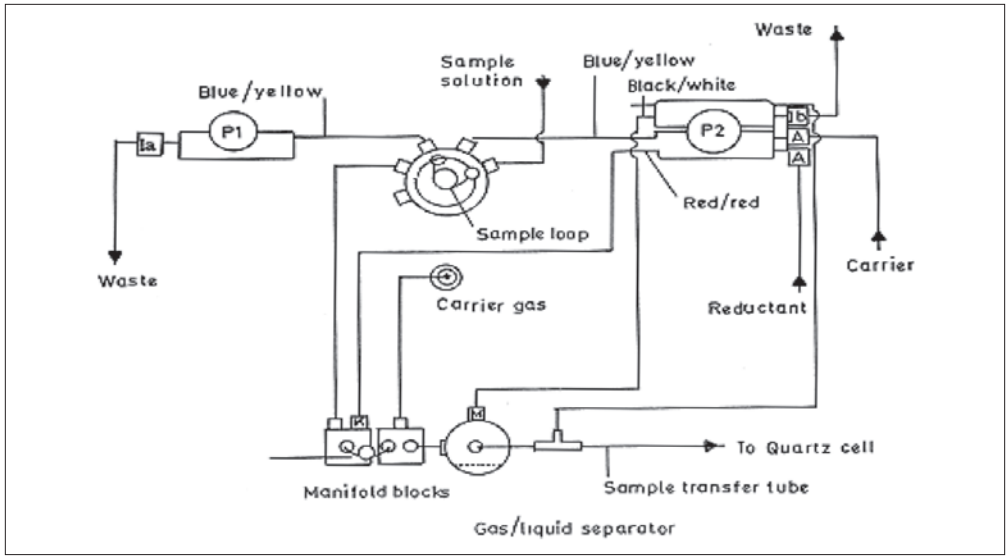


*Figure 1.* A modified tubing arrangement for tin analysis with FIAS-400.

***Table 1.*** **Recommended Conditions for Tin Analysis Using FIAS-400.**

| **Element: Sn**<br>**Signal Type: AA** | | **Wavelength (nm): 286.3**<br>**Technique: FIAS-MHS** | | **Slit (nm): 0.7**<br>**Read Time: 15 seconds** | |
|---|---|---|---|---|---|
| **Step** | **Time (Sec)** | **Pump 1 Speed** | **Pump 2 Speed** | **Valve Position** | **Read Step** |
| Pre-fill | 15 | 100 | 120 | Fill | |
| 1 | 10 | 100 | 120 | Fill | |
| 2 | 15 | 0 | 120 | Inject | X |

– Cell Temperature: 900 °C
– Sample Volume: 500 µL
– Replicates: 3

Analysis was carried out using a PerkinElmer AAnalyst™ 300 (comparable results could be obtained using the AAnalyst 200/400, AAnalyst 700/800 or PinAAcle™ 900F/900H/900T). Data were obtained using peak height measurement for 15 second periods.

## Reagents

Carrier solution: Saturated $H_3BO_3$ (approx. 50 g/L) in 1.0% (v/v) HCl.

Reductant: 0.4% $NaBH_4$ in 0.05% NaOH.

Sample Diluent: Saturated $H_3BO_3$ (approx. 50 g/L) in 1.0% (v/v) HCl.

## Measurement

The spectrometer was calibrated with prepared tin reference solutions of concentrations 5, 10 and 20 µg/L. A typical calibration curve is shown in Figure 2 and a typical atomization signal is displayed in Figure 3.

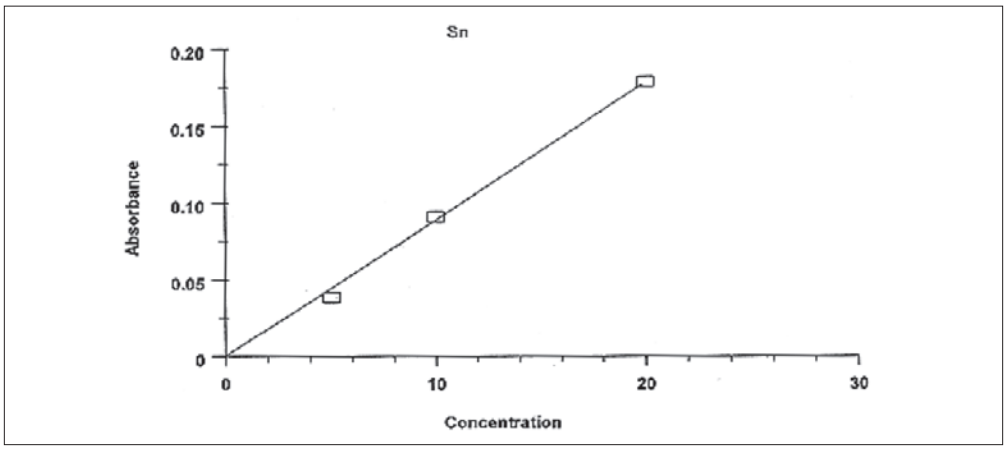


*Figure 2.* Calibration function for measurement of tin using FIAS-400. The reference solution contains concentrations of 5, 10 and 20 µg/L.

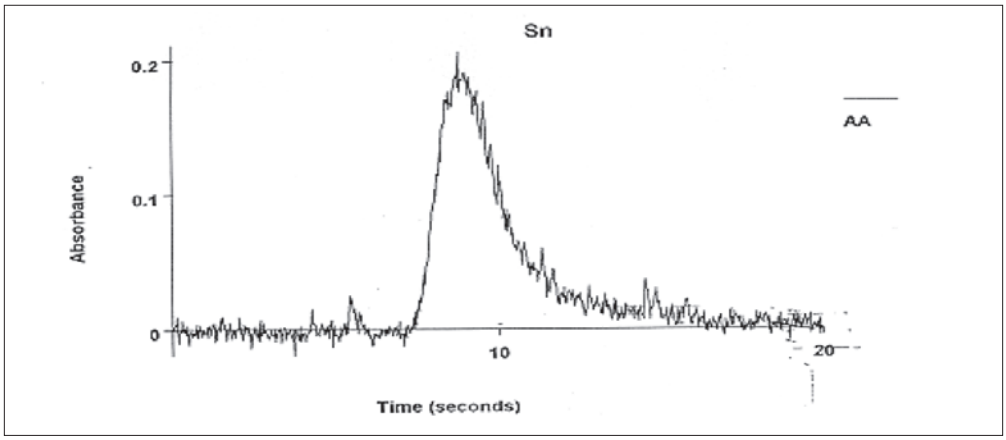


*Figure 3.* Atomization signal of tin in bead-wire coating.

## Results

Using the method described above, samples of bead-wire coating were analyzed and the results are displayed in Table 2. Bead-wire coating sample 2 was spiked with 5 µg/L Sn and resulted in a recovery of 98% (Table 3).

***Table 2.*** **Tin Content of Bead-wire Coating Samples.**

| Sample | Tin Content (µg/L) | Relative Standard Deviation (n=3) (%) |
|---|---|---|
| Bead-wire coating 1 | 9.3 | 0.5 |
| Bead-wire coating 2 | 9.6 | 1.5 |

***Table 3.*** **Results of Spiked Bead-wire Coating Samples.**

| Sample | Spiked amount (µg/L) | Recovery (%) |
|---|---|---|
| Bead-wire coating 2 | 5.0 | 98 |

## Conclusion

On-line introduction of small amounts of air (oxygen) into the quartz cell significantly improves the tin signal. This modified design is very simple and helps in activating the quartz cell much faster than conventional treatment with hydrofluoric acid.

## References

1. "Recommended Analytical Conditions and General Information for Flow Injection Mercury/Hydride Analyses Using the PerkinElmer FIAS-100/400" Technical Note, PerkinElmer.

## Acknowledgement

Bead-wire samples were obtained from Hari Sankar Singhania Elastomer & Tyre Research Institute (HASETRI), P.O. Tyrefactory, Dt. Rajsamand, Rajasthan 313342.

**PerkinElmer, Inc.**
940 Winter Street
Waltham, MA 02451 USA
P: (800) 762-4000 or
(+1) 203-925-4602
**www.perkinelmer.com**



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