

Evaluation of a New Forked Platform Design for Graphite Furnace AAS

Application Note

Atomic Absorption

Authors

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Introduction

Graphite furnace techniques are characterized by excellent analytical sensitivity. They are methods of choice for the determination of trace elements in various environmental, biological, and clinical samples. However, they can be plagued by chemical and background interferences which complicate methods development and sample analysis. Using the graphite platform technique, these interferences can be reduced. The graphite platform is placed in the graphite tube. Sample atomization takes place from the platform rather than the tube wall. The temperature of the platform lags behind the temperature of the tube wall and the gaseous environment. This temperature lag causes delayed sample vaporization and analyte atomization. This can reduce chemical and background interferences.

This application note describes the evaluation of a new forked platform design for the Agilent GTA graphite furnaces. The forked platform is installed into a partitioned tube. The partitions are "notched" and the platform is designed to "catch" in the partition notches. This provides minimal physical contact between the platform and tube wall. Platform rotation is prevented during insertion of the tube into the graphite furnace. The tube and platform are shown in Figure 1. Advantages include; simple positioning of the tube within the furnace, easy alignment of the sample dispensing capillary tip, (relative to the platform) and stability of the platform during operation. Another advantage is that the platform can be removed and the same tube design can be utilized for wall atomization.



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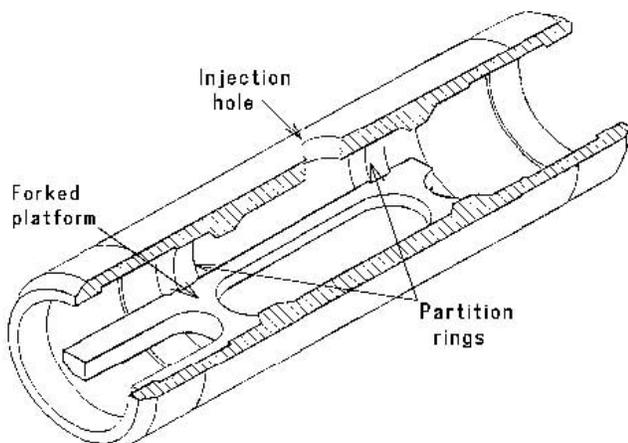


Figure 1. Tube and platform.

The results of an evaluation of this platform are reported below. The determination of five elements (As, Se, Pb, Cd, and Sn) was used to examine the following performance characteristics:

- The observed thermal delay, in comparison with current Varian platforms
- The accuracy of results for an EPA standard
- The analytical results of a difficult environmental sample, in comparison with current Varian platforms

Methodology

Agilent SpectrAA-400 Zeeman and SpectrAA-400 Deuterium spectrometers were used for this evaluation. The Zeeman spectrometer was used for the Cd and Sn measurements and the deuterium spectrometer for the As, Se, and Pb measurements. Table 1 lists the instrument operating parameters. Hollow cathode lamps were utilized for this study. The furnace parameters are summarized in Table 2. The maximum atomization ramp rate was used for all elements. The modifier solutions used in this study are listed in Table 3.

Table 1. Instrument Operating Parameters

Element	As	Se	Cd	Pb	Sn
Wavelength	193.7	196.0	228.8	283.3	286.3
SBW	0.5	1.0	0.5	0.5	0.5
Lamp current	10	12	4	5	7
Measurement mode	Peak area	Peak ht.	Peak area	Peak area	Peak area

Table 2. Furnace Parameters

Element	As	Se	Cd	Pb	Sn	
Dry (all elements)		Hot inject				
		Temperature 125 °				
		Injection rate 9				
Ramp to	350 °					
Time	40 s					
Ash	900 °	650 °	700 °	1000 °	1000 °	
Atomize	2550 °	2500 °	2300 °	2300 °	2600 °	

Table 3. Matrix modifiers

Element	Matrix modifier
As	1000 mg/L Ni in 5% HNO ₃
Se	1000 mg/L Ni in 5% HNO ₃
Cd	1% H ₃ PO ₄
Pb	1% H ₃ PO ₄
Sn	200 mg/L Pd, 2% Citric Acid

Results

The evaluation of the new platform began with the observation of the thermal delay. This was then compared to the delay obtained with current platforms. The results of this investigation are shown in Figures 2 and 3. In the case of Pb, Cd, and Se (Figure 2), the observed thermal delay was the same as that obtained with the current platform. The absorbance signals for Pb and Cd were essentially identical. With Se the thermal delay was very similar, but the sensitivity in peak height was greater for the regular platform. Peak area measurements were within 10%. This slight difference in sensitivity could be expected in the normal variability between graphite components.

In the absorbance measurements for As and Sn the use of the forked platform resulted in an earlier atomization signal. However, peak area sensitivities with the forked platform were within 10% of those obtained for the regular platform. A number of factors influenced this difference in observed thermal delay. In particular:

- The age of the graphite tubes
- The temperature program

As only a small number of graphite tubes and platforms were investigated, further work would be necessary to show a consistent variability.

An EPA standard was analysed to determine the accuracy of results while utilizing the forked platform. These results are shown in Table 4. The 95% confidence level represents the mean value from the EPA's study plus or minus two standard deviations. The results from the evaluation of the forked platform fall within this range in all cases.

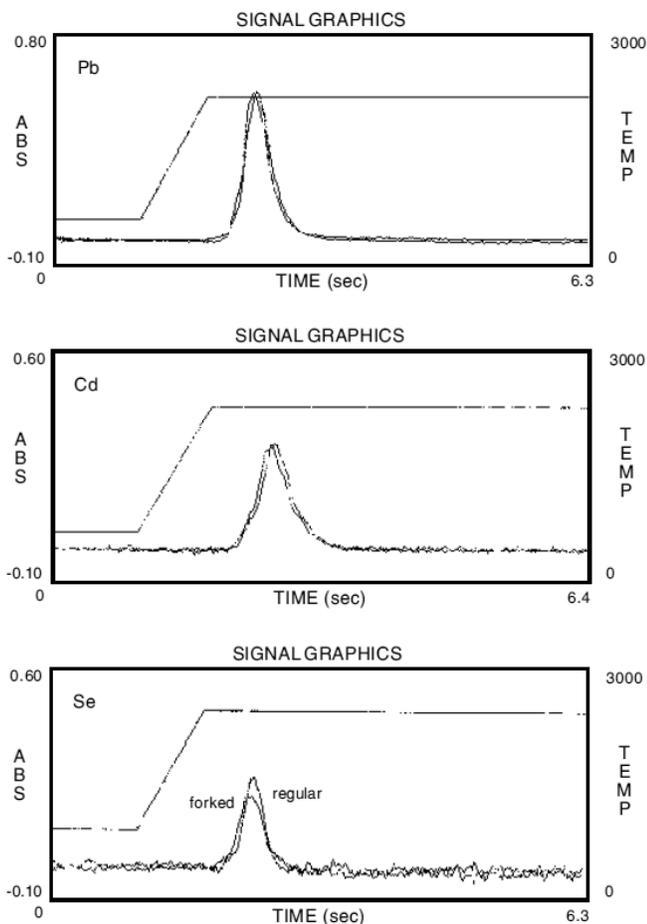


Figure 2.

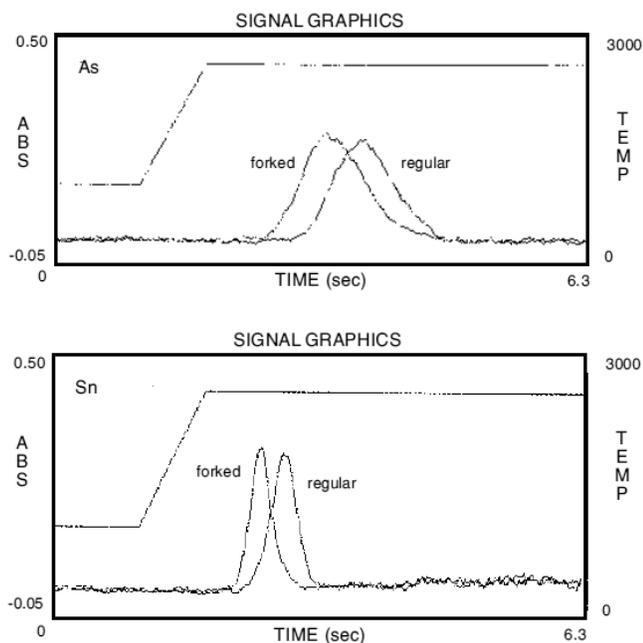


Figure 3.

Table 4. Results for EPA Trace Metals Quality Control Reference Standard

Element	"True" value	X	95% Confidence level	This study (Forked platform)
As	50.0	49.6	38.9–60.3	51.2
Se	50.0	45.8	36.1–55.5	50.1
Cd	4.9	4.58	3.30–5.86	5.5
Pb	50.0	50.4	40.3–60.5	47.7

In the final evaluation of the forked platform, 5 elements were determined in a difficult, digested environmental sample. The results were compared to those obtained with the regular platform. As shown in Table 5 there was no significant difference between results obtained with the forked platform and those obtained with the current platform design.

Table 5. Results for a Difficult Environmental Sample

Element	Forked platform	Regular platform
As (1g/L)	88.0	94.0
Se (1g/L)	<1.0	<1.0
Cd (1g/L)	13.0	12.8
Pb* (1g/L)	4.9	5.1
Sn (1g/L)	184.0	189.0

*Sample diluted 1:100 for Pb determination.

Conclusion

The forked platform as described in this application note has several advantages over other designs:

The stability and excellent precision.

In terms of performance, there was little difference between the forked platform and the current design. Although there was some variation in the observed thermal delay for two of the elements, sensitivities and results for the determination of various elements in an EPA standard and difficult environmental samples were identical.

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