



# Simultaneous trace metal determinations in soil using SIMAA 6000 AAS

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Trace metal determinations in soil are used to evaluate contamination or to monitor the extent and success of remediation efforts. The elements measured can vary widely, but typically include the toxic elements As, Se, Tl, and Pb. These elements may occur naturally in soil and sediment, but the native levels are generally low. Larger concentrations are generally a result of contamination from industrial processes or as a result of improper waste disposal. Several measurements may be required to map a site or to monitor the course of remediation, and increased productivity can be realized by measuring several elements simultaneously at the low levels required. This note describes the analysis of a variety of sediments and soils for four elements measured simultaneously using graphite furnace atomic absorption and Zeeman background correction.

## Experimental

The Perkin-Elmer SIMAA™ 6000 atomic absorption system (AAS) was used for all measurements. The SIMAA uses an echelle spectrometer to direct light onto a solid-state diode array detector for up to six elements at once. The transversely heated graphite furnace and Zeeman background correction are important components of the stabilized temperature platform furnace concept and contribute to the overall freedom from interferences. The AAS was set

up as described in Table 1 for As, Se, Tl, and Pb. The furnace program used for all elements is shown in Table 2.

The samples examined were certified samples of estuarine sediment, river sediments, and soils from High Purity Standards, Charleston, SC. The materials were prepared in 2% nitric acid and provide a variety of contaminant levels and natural elemental composition levels.

The method used for analysis followed the protocol described in U.S. EPA method 200.9<sup>1</sup>. The method uses

Table 1. Instrument Parameters

Perkin-Elmer SIMAA 6000 AAS				
Element	As	Se	Tl	Pb
Wavelength (nm)	193.7	196.0	276.8	283.3
Lamp	EDL	EDI	HCL	HCL
Integration time (sec)	5	5	5	5
Std 1 (µg/L)	15	15	15	15
Std 2 (µg/L)	45	45	45	45
Std 3 (µg/L)	150	150	150	150
Calibration Method	Zero-intercept, nonlinear			
Technique	Zeeman background correction			
Signal Processing	Peak area			
Replicates	3			
Sample Volume	20 µL			
Chemical Modifier	5 µL of 0.005 mg Pd and 0.003 mg Mg(NO <sub>3</sub> ) <sub>2</sub>			

STPF concepts for 16 elements and is well suited for use with a simultaneous AAS. The method uses an alternate gas, 5% hydrogen in 95% argon, during the dry and pyrolysis pretreatment steps to ensure the complete removal of Cl, resulting from native occurrence in the sample or added during the digestion procedure. A step is added to the furnace program to flush the furnace, removing residual hydrogen before the atomization step.

## Results and Discussion

The calibration curves are shown in Figure 1. They are linear to approximately 150 µg/L, but can be used to higher concentration successfully if allowed by regulatory authorities. Observation of the calibration curve and QC checks over the full concentration range will ensure that the data meet the quality objectives. The estimated instrument detection limits are shown in Table 3 and have been calculated by multiplying by three

the standard deviation of 7 replicates of a low-level standard. This detection limit is reflective of the instrument performance and may vary if digestion procedures introduce more variation. The detection limits are shown both for the solution introduced into the instrument and what may be detected in soil using a typical digestion of 1 g of soil digested and diluted to a final volume of 100 mL.

Table 2. THGA Program

Step	Temp (°C)	Ramp Time (s)	Hold Time (s)	Internal Gas Flow (mL/min)	Gas Type	Read Step
1	110	1	30	250	Ar/H <sub>2</sub>	
2	140	15	30	250	Ar/H <sub>2</sub>	
3	800	10	20	250	Ar/H <sub>2</sub>	
4	200	1	30	250	Ar	
5	2150	0	5	0	Ar	X
6	2450	1	3	250	Ar	

The injection temperature was 110°C.

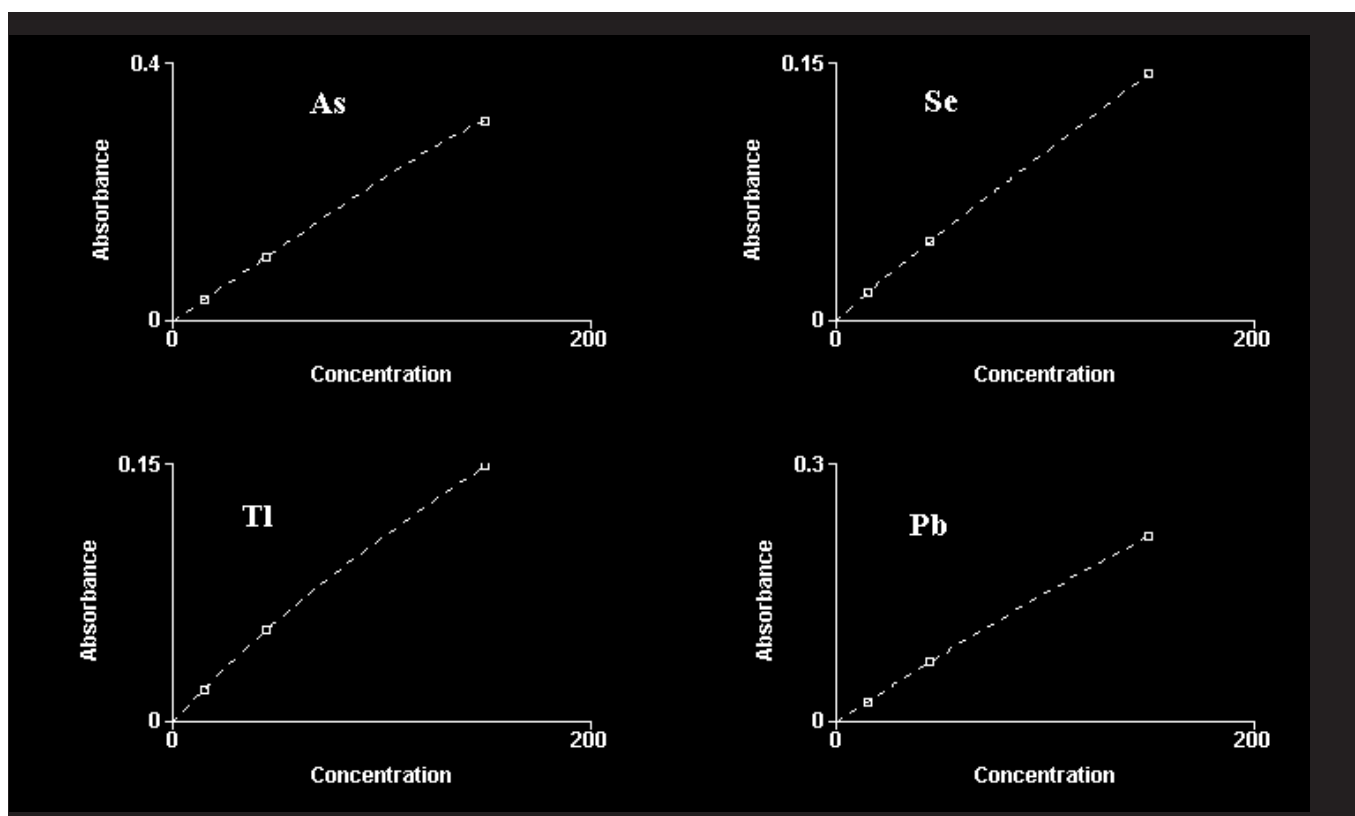


Figure 1. Calibration curves.

**Table 3. Estimated Detection Limits**

Element	Solution Detection Limit (µg/L)	Soil Detection Limit (mg/kg)
As	1	0.1
Se	0.3	0.03
Tl	0.8	0.08
Pb	0.5	0.05

The peaks from spiked River Sediment A are shown in Figure 2. Note that the background signal is high, but can be easily compensated with Zeeman background correction.

The results obtained here are compared with the certificate values in Tables 4-8. The dilutions varied somewhat depending on the extent of contamination and 0-2x was found to be generally applicable for moderate

contamination. In the case of higher contamination the software can be directed to automatically perform additional dilutions to bring the absorbance to a value within the calibration range. Spikes were performed at 25 µg/L on the samples (diluted by the instrument in some cases) and excellent recoveries were observed.

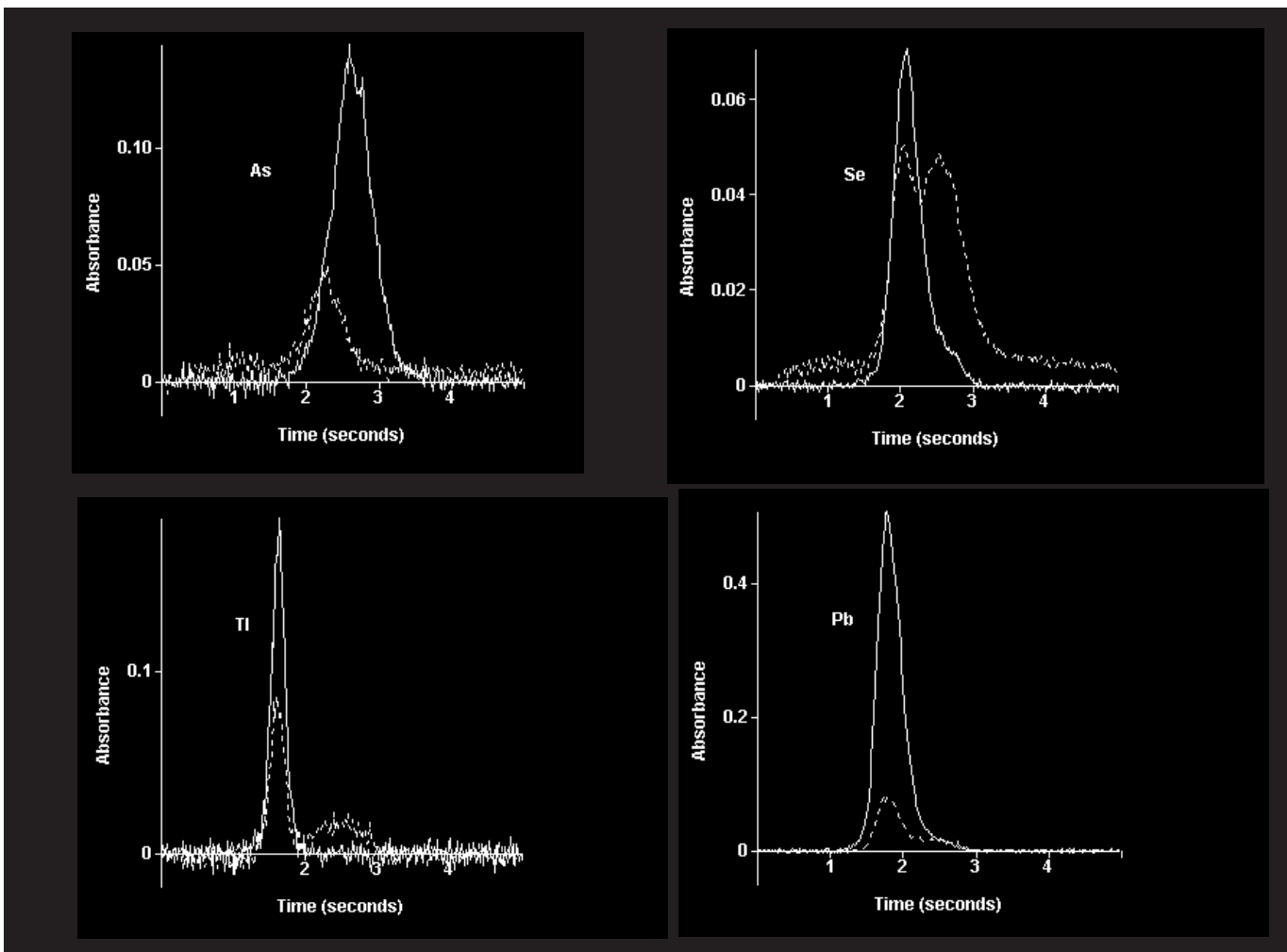


Figure 2. Atomic peaks (solid) and background peaks (dotted) from spiked River Sediment A.

**Table 4. River Sediment A Results**

Analyte	Certified Value (µg/L) ± 0.5%	Average Results (SD) (µg/L)	Recovery of Certified Value (%)	Recovery of Spike (%)
As	600	583 (4)	97.2	106
Se	20	23.4 (0.7)	117	95.5
Tl	10	10.4 (0.1)	104	103
Pb	7000	6360 (15)	90.9	113

**Table 5. River Sediment B Results**

Analyte	Certified Value (µg/L) ± 0.5%	Average Results (SD) (µg/L)	Recovery of Certified Value (%)	Recovery of Spike (%)
As	200	210 (7)	105	114
Se	10	11.5 (1.3)	115	97.0
Tl	10	8.6 (0.7)	86.0	101
Pb	2000	1890 (36)	94.5	88.1

**Table 6. Soil A Results**

Analyte	Certified Value (µg/L) ± 0.5%	Average Results (SD) (µg/L)	Recovery of Certified Value (%)	Recovery of Spike (%)
As	150	146 (4)	97	109
Se	10	9.6 (0.5)	96.0	86.7
Tl	–	<DL	–	98.8
Pb	400	385 (2)	96.3	98.5

**Table 7. Soil B Results**

Analyte	Certified Value (µg/L) ± 0.5%	Average Results (SD) (µg/L)	Recovery of Certified Value (%)	Recovery of Spike (%)
As	6000	6546 (71)	109	97.4
Se	–	6.9 (0.3)	–	91.5
Tl	–	<DL	–	96.0
Pb	60 000	61 800 (130)	103	95.9

**Table 8. Estuarian Sediment Results**

Analyte	Certified Value (µg/L) ± 0.5%	Average Results (SD) (µg/L)	Recovery of Certified Value (%)	Recovery of Spike (%)
As	100	86.7 (0.6)	86.7	86.7
Se	50	49.3 (1)	98.6	88.2
Tl	–	<DL	–	85.7
Pb	300	289 (0.5)	96.3	101

## Conclusions

The simultaneous determination of As, Se, Tl, and Pb in soil digests has been demonstrated to be analytically feasible. The detection limits for the four elements are excellent and show that very little compromise is necessary in combining elements, even those with differing volatilities. The combination of transversely heated furnace, Zeeman background correction, and the other STPF elements offered in the system reduce interferences, reducing the requirement for the method of standard additions.

## Reference

1. EPA Method 200.9, The Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrometry, Version 2.2, in Methods for the Determination of Metals in Environmental Samples, Supplement I, EPA/600/R-94/111, (May 1994).

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