

## Enhanced Detection of Trace Elements Using a Microwave Plasma Atomic Emission Spectrometer (MP-AES) Coupled with an Ultrasonic Nebulizer

### INTRODUCTION

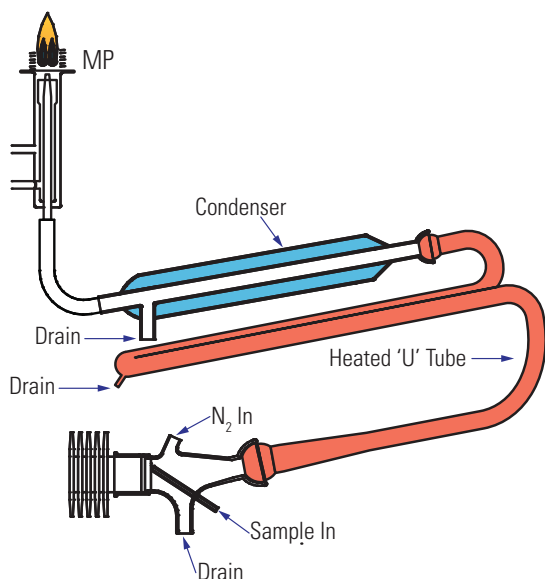
The Agilent 4100 MP-AES is a new atomic emission spectrometer that features a microwave plasma as the emission source. This source is sustained with nitrogen gas, avoiding the need for more expensive flammable and oxidizing gases used with traditional flame atomic absorption (AA) spectrometers. In addition, the Agilent 4100 MP-AES features a scanning spectrometer with a CCD detector for multielement measurements, providing sub-ppb detection limits for many elements.

This technical note will examine the coupling of a commercial ultrasonic nebulizer (USN) to the Agilent 4100 MP-AES. The USN is commonly used with argon ICP-AES (inductively coupled plasma atomic emission spectrometer) instruments for enhanced element detection limits versus conventional pneumatic nebulizers. As the 4100 MP-AES is also supplied with a pneumatic nebulizer, improved trace element detection should also be possible with the USN. Figures of merit will include calibration and instrument detection limits (IDLs). Of particular interest is improved detection of more difficult elements such as arsenic and selenium and platinum group elements (PGEs) such as platinum, palladium, and gold.

### INSTRUMENTATION

MP-AES: **Agilent 4100**

Ultrasonic Nebulizer (USN): **CETAC U5000AT<sup>+</sup>**



Agilent 4100 MP-AES with CETAC U5000AT<sup>+</sup> Ultrasonic Nebulizer

CETAC U5000AT<sup>+</sup> Schematic: The U5000AT<sup>+</sup> consists of a piezoelectric transducer, a heated u-tube, and an electrothermally cooled condenser.

**Operating Conditions – 1. Standard Nebulizer**

MP-AES:	Agilent 4100
Gas Supply	Nitrogen
Nebulizer Gas Pressure:	100 to 240 kPa
Solution Uptake:	1.4 mL/min (pumped)
Pump Tubing:	1.02 mm i.d. PVC
Nebulizer:	Glass Concentric
Spray Chamber:	Glass Cyclonic
Read Times:	10 s; 30 s for As and Se
Replicates:	7
Background correction:	Auto

**Operating Conditions – 2. Ultrasonic Nebulizer**

Nebulizer System:	CETAC U5000AT <sup>+</sup>
Gas Supply:	Nitrogen
Nebulizer Gas Pressure:	125 to 240 kPa
Heater Temp:	140°C
Cooler Temp:	3°C
Solution Uptake:	0.85 mL/min (pumped)
Pump Tubing:	0.76 mm i.d. PVC
Read time:	10 seconds (all elements)
Replicates:	7
Background correction:	Auto

**Experimental Parameters – I****Reagents:**

- Nitric Acid, Baker Instra-Analyzed Reagent, J.T. Baker, Phillipsburg N.J. USA
- Hydrochloric Acid, BDH Aristar Plus, VWR, Radnor, PA USA
- Agilent Technologies QCSTD-27 multielement standard
- Various single-element standards, Inorganic Ventures, Christiansburg, VA USA

**Blank and Standards Preparation:**

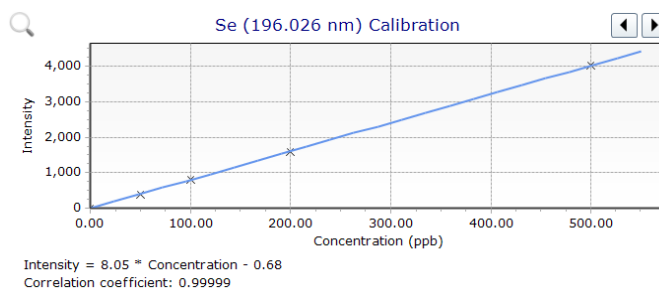
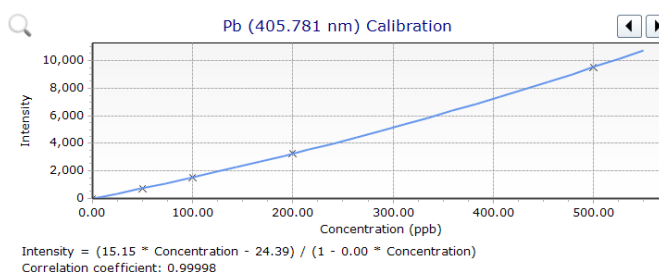
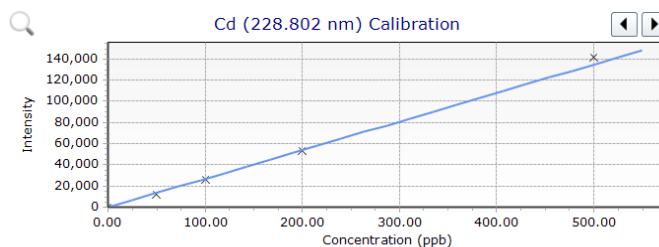
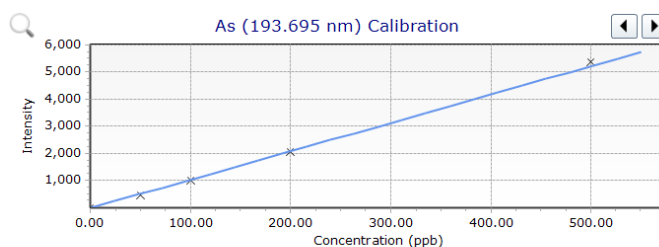
Reagent blanks and calibration standards were prepared by weight in low density polyethylene (LPDE) bottles in 1.0 % (v/v) nitric acid or 1% (v/v) hydrochloric acid (for PGEs: Au, Ir, Rh, Ru, Pd, Pt). Standard concentrations were 50, 100, 200, and 500 µg/L.

**Experimental Parameters – II****USN Setup, Calibration, and Instrument Detection Limits:**

The standard nebulizer and spray chamber were first removed from the Agilent 4100 MP-AES. Using an interface kit, the ultrasonic nebulizer was connected to the MP-AES via a nebulizer gas line and a sample out line. The nebulizer gas line contained a flow restriction fitting and the sample out line a 12/5 glass adapter for direct connection to the torch. Sample solution flow to the ultrasonic nebulizer transducer was from the host MP-AES peristaltic pump. Setup of the ultrasonic nebulizer took only 5 minutes.

The reagent blank and the four calibration standards were then introduced to the MP-AES with the ultrasonic nebulizer. Instrument detection limits (IDLs) are calculated as 3 times the standard deviation of the blank concentration. Note that data was obtained under non-cleanroom conditions.

Calibration curves for As, Cd, Pb, and Se are given below; correlation coefficients range from 0.9997 to 0.9999.



## INSTRUMENT DETECTION LIMIT COMPARISON

The ultrasonic nebulizer lowers instrument detection limits (IDLs) for 15 of the 20 elements (listed below) by a factor 2.0 to 100, with very significant reductions (10x or more) for Al, As, Au, Cd, Pt, and Se. Some elements have little or no reduction in IDL, which may be due to a more complex background spectrum and/or elevated blanks. All but four of the elements (As, Co, Pd, Th) in the comparison table have IDLs below 1 µg/L.

Element	Wavelength (nm)	Std. Neb.* IDLs (µg/L)	USN IDLs (µg/L)	Factor
Ag	328.068	0.5	0.2	2.5
Al	396.152	0.6	0.05	12
As	193.695	45	1.2	37
Au	267.595	1.8	0.1	18
Be	234.861	0.1	0.09	1.1
Cd	228.802	1.4	0.07	20
Co	340.511	3.1	2.9	1.0
Cr	425.433	0.5	0.3	1.6
Cu	324.754	0.6	0.6	1.0
Fe	259.940	1.6	0.2	8.0
Mn	403.076	0.25	0.12	2.0
Mo	379.825	1.5	0.5	3.0
Ni	352.454	1.3	0.4	3.3
Pb	405.781	4.4	0.9	4.8
Pd	340.458	3.8	1.2	3.1
Pt	265.945	4.5	0.3	15
Se	196.026	70	0.7	100
Th	401.913	6.0	3.7	1.6
Tl	535.046	2.1	0.3	7.0
Zn	213.857	2.8	0.7	4.0

\*These limits are from *Agilent 4100 MP-AES Typical Performance* document.

## ADDITIONAL INSTRUMENT DETECTION LIMITS

Instrument detection limits obtained with the ultrasonic nebulizer and the MP-AES are listed for six additional elements in the table below, including iridium, antimony, and uranium. The detection limit for antimony is notably low at 0.4 µg/L.

Element	Wavelength (nm)	USN IDLs (µg/L)
Ir	263.971	1.6
Rh	343.489	2.5
Ru	372.803	0.6
Sb	231.147	0.4
U	434.169	1.7
V	309.311	0.6

## SUMMARY

Use of an ultrasonic nebulizer with the Agilent 4100 MP-AES enables significantly lower instrument detection limits (greater than 10 fold reduction) for a number of important elements such as Al, As, Au, Cd, Se, and Pt. Other elements such as Ag, Cr, Cu, Ni, Pb, and Tl have a lower range of improvement (factor of 1 to 7); this lower range may be due to higher instrument blanks and more complex background spectra. Use of higher purity nitric and hydrochloric acids may lower instrument blanks.

Instrument detection limits obtained with the ultrasonic nebulizer were all measured at a 10 second read time. Longer read times and optimized gas pressures (for individual elements) may provide further improvement in detection limits.