

# Agilent's Atomic Spectroscopy Portfolio

**ICP-OES**



**4100 MP-AES**

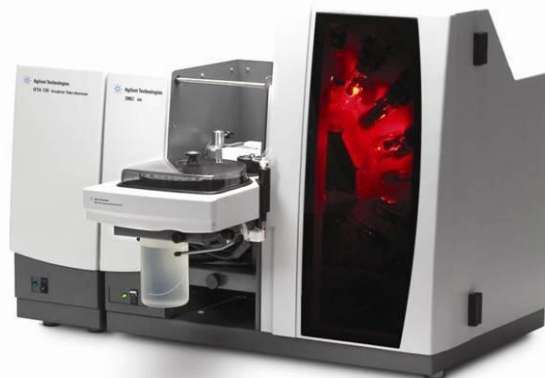


**Flame AAS**



AAS instruments can be flame only, furnace only, or combined (switchable)

**Graphite Furnace AAS**



# Lower Operating Costs, Safer Operation, More Productive. Agilent 4100 MP-AES



**Wayne Blonski**  
**Agilent Technologies**

# Common Challenges facing Laboratories doing Elemental Analysis Today

- Increased need for multi-element determination over a wide dynamic range
- Desire to reduce the overall cost of analysis due to rising costs (instrument supplies and consumables; power; labour etc)
- Difficulty in sourcing some gases – esp. in remote areas and emerging geographies
- Availability of suitably trained personnel to develop methods, perform sample measurement and interpret results
- Some laboratories under pressure to improve safety by removing flammable gases



# Agilent 4100 Microwave Plasma-Atomic Emission Spectrometer (MP-AES)

New technique for elemental determination using atomic emission

- Microwave excited plasma source
- Nitrogen based plasma - runs on air (using a N<sub>2</sub> generator)

## Improved performance compared with flame AA:

- Higher sample throughput with fast sequential measurement
  - More than 2x faster than conventional flame AA
- Superior detection limits and improved dynamic range

## Easy to use:

- New generation software featuring automated optimization and software applets that load a preset method
- One piece torch with easy torch removal and replacement – no alignment

## Reduced operating costs:

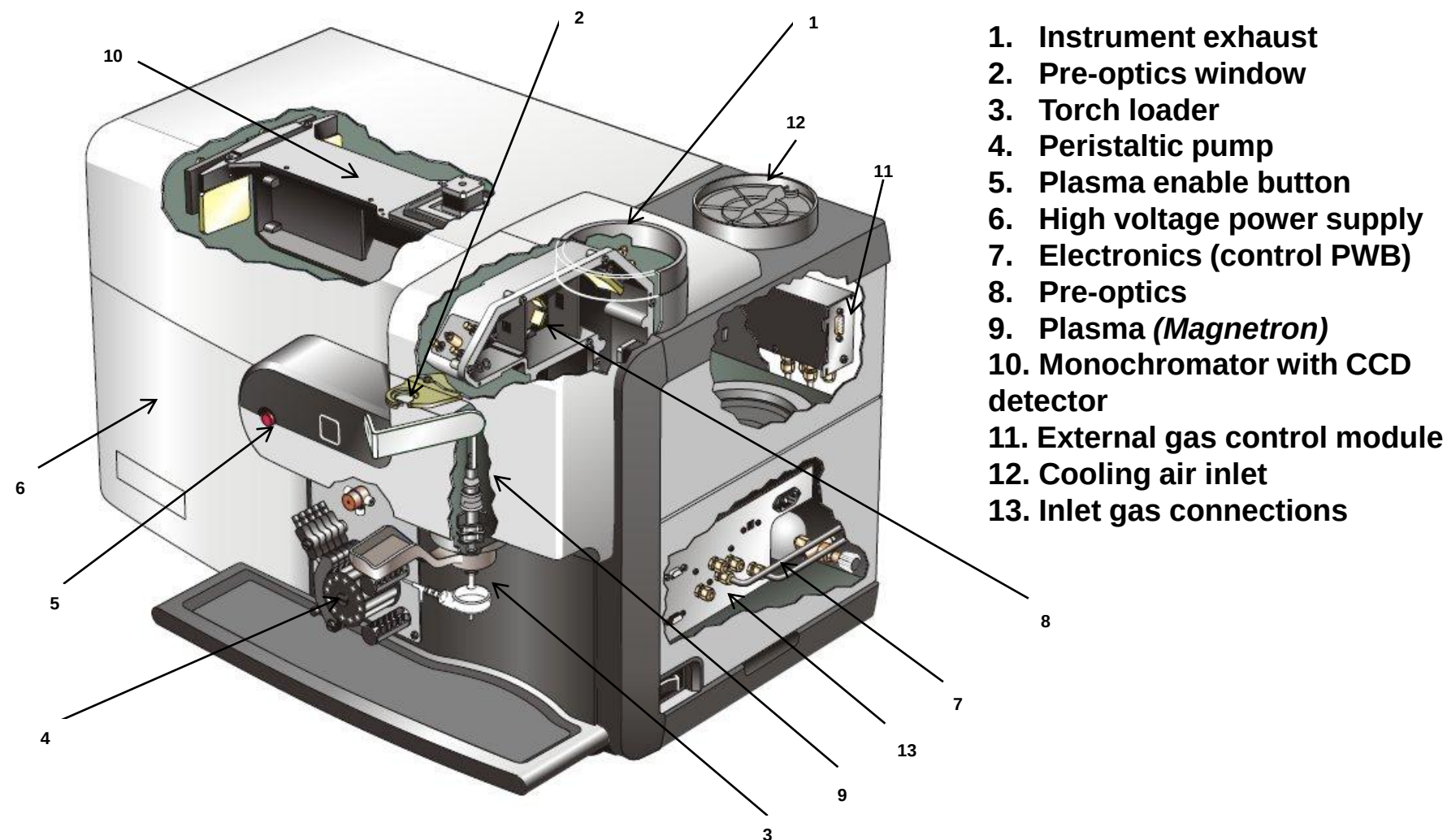
- Runs on air – eliminates need for Acetylene, Argon, etc.
- Eliminates need for source/hollow cathode lamps
- Simple installation – no chiller, 10 A supply

## Improved Safety:

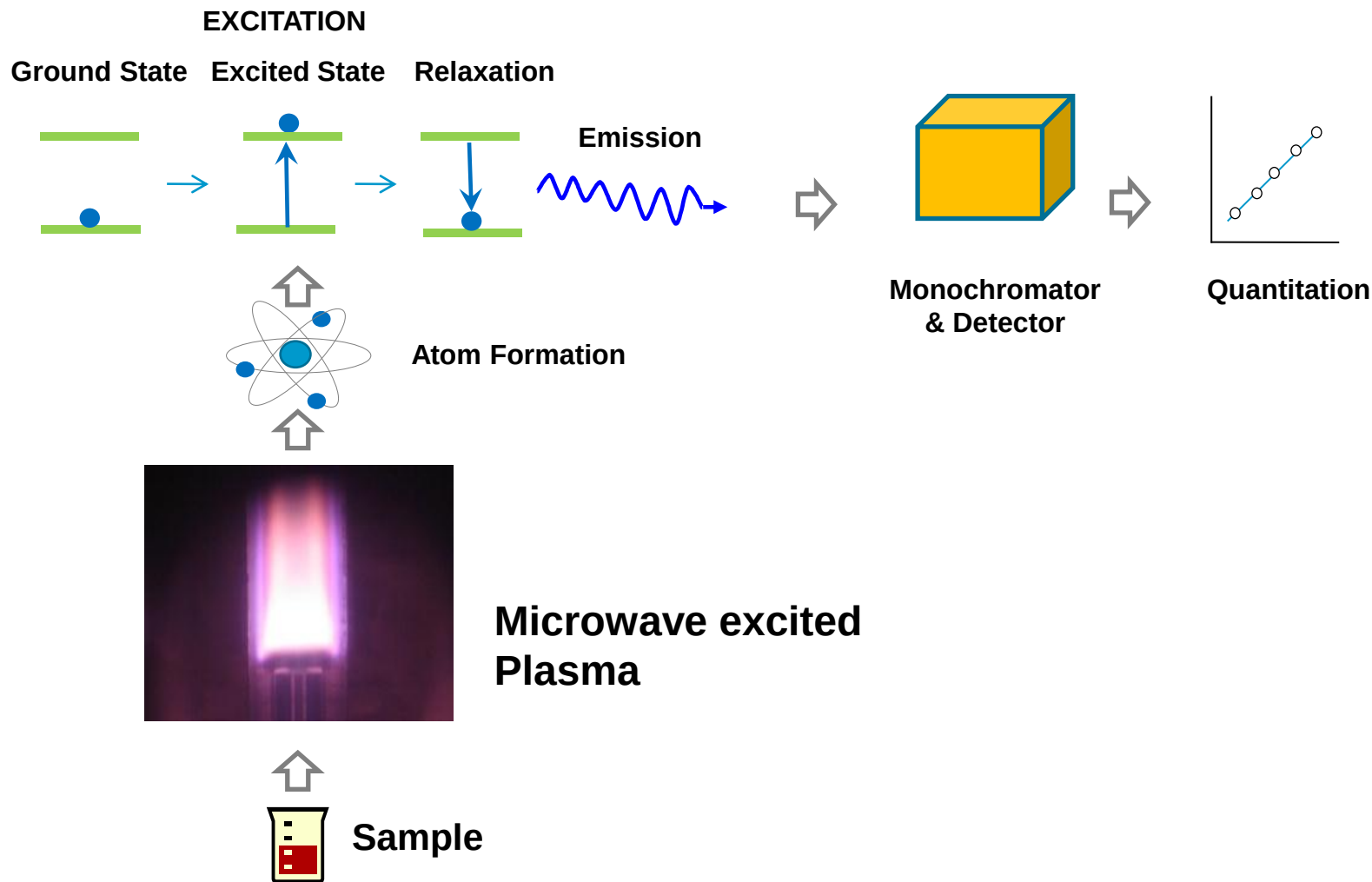
- Eliminates need for flammable gases and cylinder handling
- Safe, reliable unattended multi-element overnight operation



# Schematic Diagram



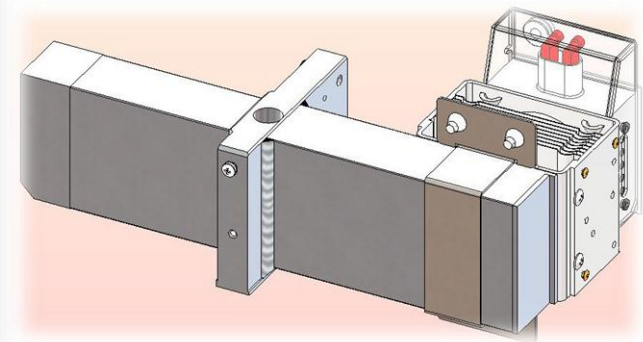
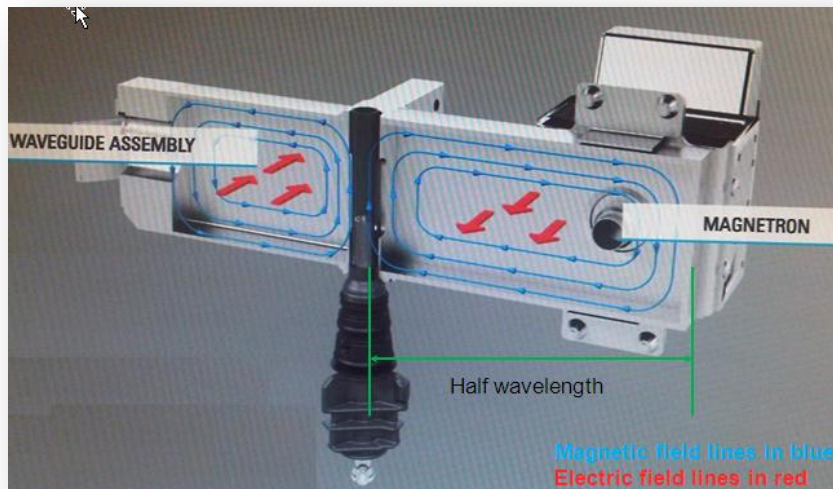
# Microwave Plasma Emission Overview



# How Does MP-AES Work?

## Agilent's patented microwave waveguide technology

- Using nitrogen as the plasma gas gives a robust plasma with a conventional torch. Nitrogen can be supplied either via bottled gas or via nitrogen generator
- Magnetic excitation gives a toroidal plasma and an effective central zone for sample injection
- The microwave magnetically excited nitrogen plasma
  - Provides a robust, high temperature source in conventional torches (approx. 5000 K)
  - A cooler central channel suitable for sample atomization





# Performance – Typical Detection Limits

*DL's in ppb, clean water samples*

| Element | Flame AA | MP-AES | Element | Flame AA | MP-AES |
|---------|----------|--------|---------|----------|--------|
| K       | 0.8      | 0.65   | As*     | 60       | 45     |
| Ca      | 0.4      | 0.05   | Cd      | 1.5      | 1.4    |
| Mg      | 0.3      | 0.12   | Cr      | 5        | 0.5    |
| Na      | 0.3      | 0.12   | Mn      | 1.0      | 0.25   |
| Au      | 5        | 1.8    | Pb      | 14       | 4.4    |
| Pt      | 76       | 4.5    | Sb      | 37       | 12     |
| Pd      | 15       | 3.8    | Se*     | 500      | 70     |
| Ag      | 1.7      | 0.5    | V       | 20       | 0.2    |
| Rh      | 4        | 0.5    |         |          |        |

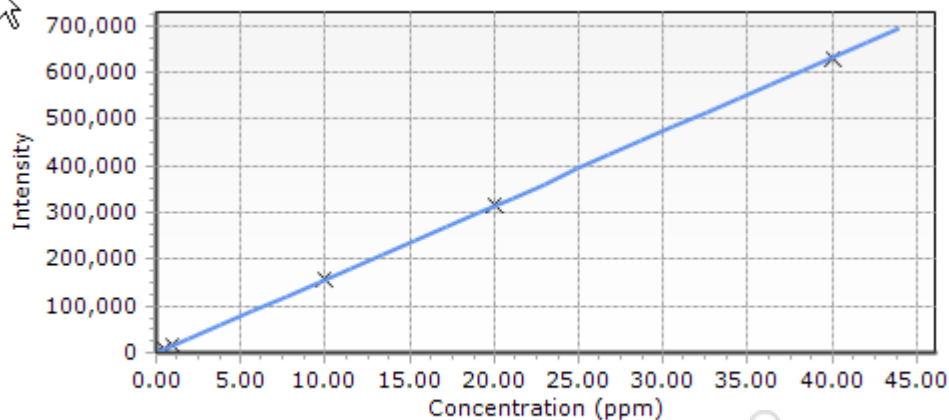
*\* 30 second integration time used for As and Se*

***3 sigma DLs using a 10 second integration time***



# Performance – Extended Working Range

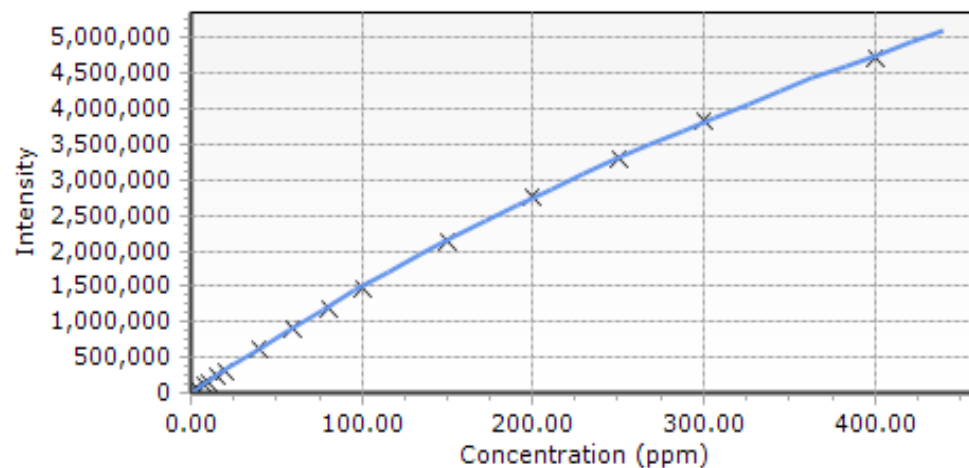
Au (267.595 nm) Calibration



Intensity = 15775.550 \* Concentration - 0.205  
Correlation coefficient: 1.00000

MP - normal AA range

Au (267.595 nm) Calibration

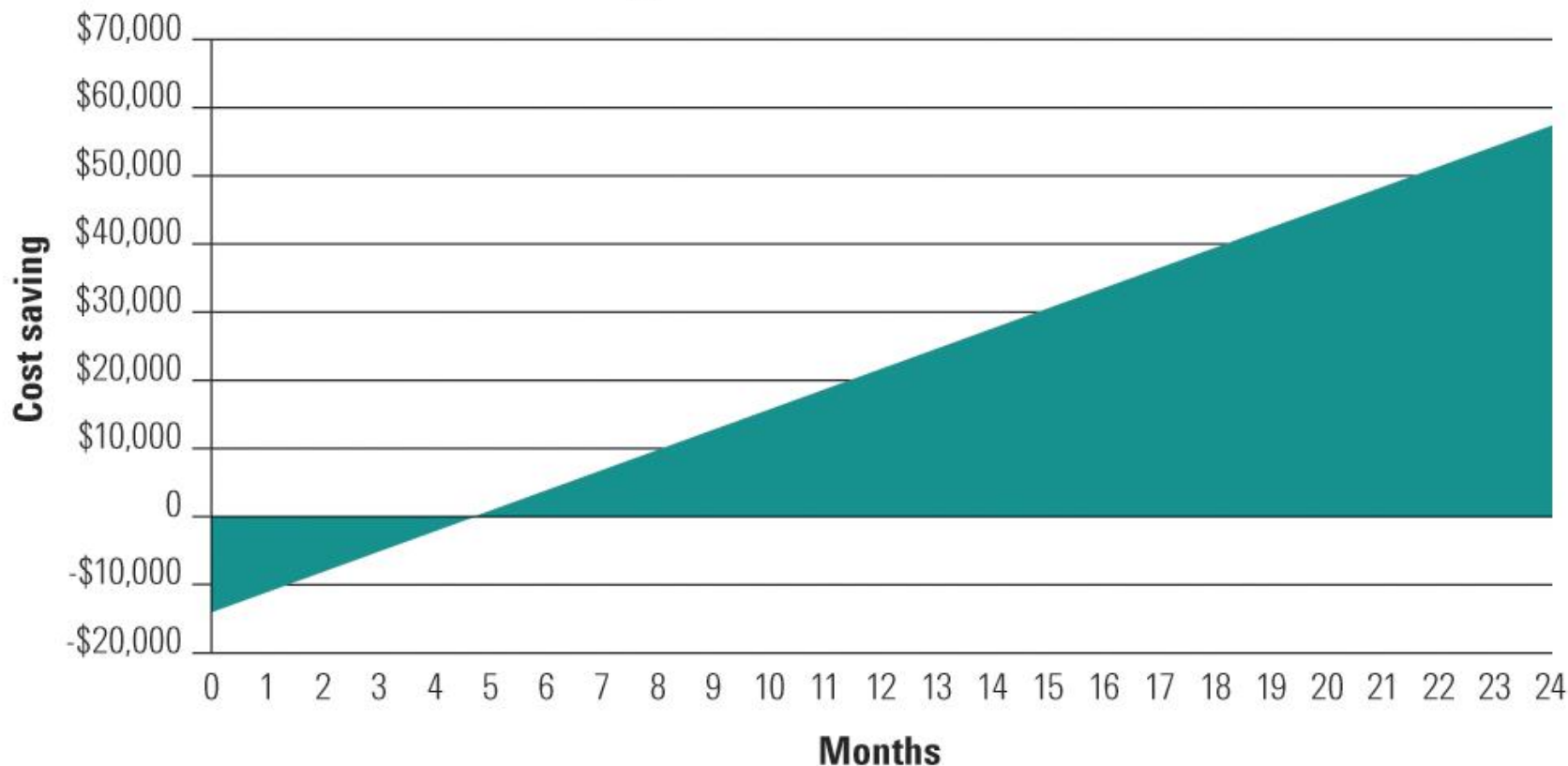


Intensity = (16408.841 \* Concentration + 0.057) / (1 + 0.001 \* Concentration)  
Correlation coefficient: 0.99998

MP - Extended Range

# Reduced Cost of Ownership

**MP-AES cost savings versus flame AA**



Cost savings based on reductions in gas expenses ONLY!.

Assumes measuring 9 elements (4 nitrous-oxide elements) in batches of 100 samples, 3 days/week using the N<sub>2</sub> generator. For a detailed cost comparison, consult your local Agilent representative



# Simple Torch Installation – No Alignment

## Torch installation in three easy steps

**1** Open the torch loader



**2** Insert the torch



**3** Close the torch loader



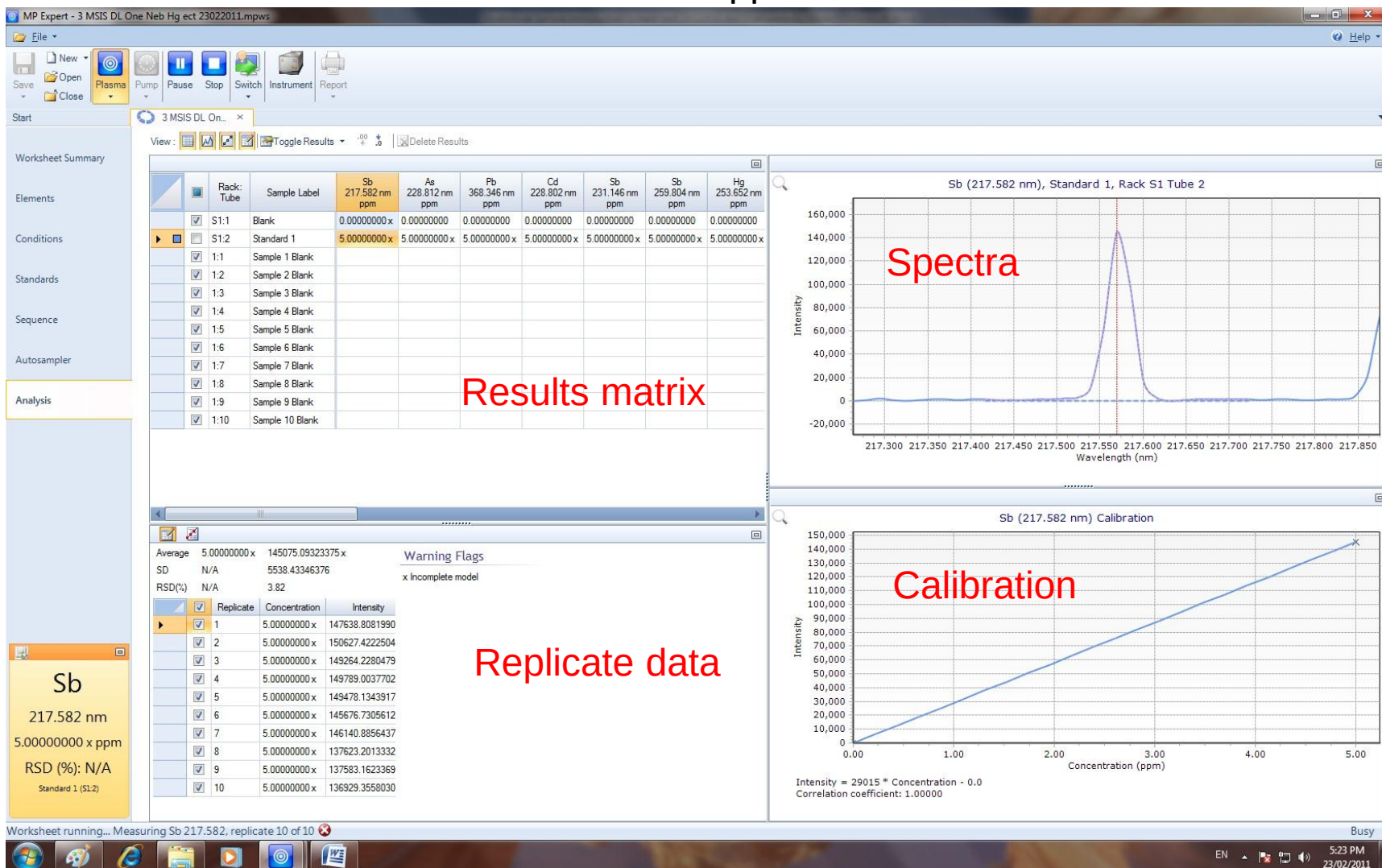
# New Generation MP Expert Software

- Windows 7, worksheet based software with significant enhancements
  - Fresh, clean look
- Provides capability for:
  - Applet style operation using preset methods, or
  - Access to full capabilities
- Automated optimization tools
- Innovative and simple to use background correction
  - No correction points to select
- Extensive help with hover tips



# New Generation MP Expert Software

- Retains familiar “worksheet” based approach



# Applet Style Operation using MP Expert Software

- Simplified software operation for operators

## Three steps to analysis

- 1 Click the icon. The applet automatically loads the pre-set method



Agriculture



Beverages



Chemicals



Energy



Food



Fuels

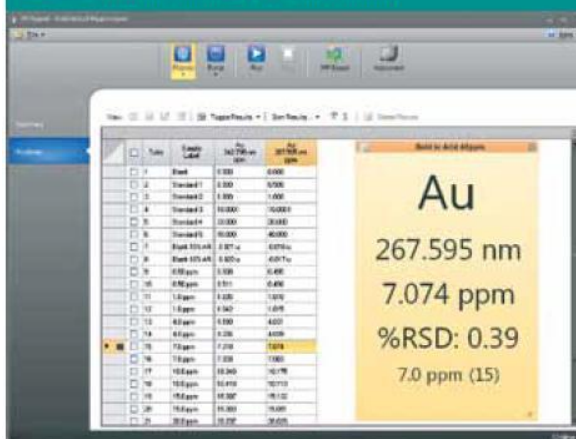


Industrial



Metals

- 2 Enter sample labels, sample type, and weight/volume correction factors



- 3 Load samples and run the analysis



Plasma



Pump



Run



Stop



MP Expert



Instrument





# Accessory Options for the 4100 MP-AES

- Automate and simplify analysis with the SPS3 autosampler (required for unattended overnight operation)
- For organic applications, use the EGCM to bleed air into the plasma minimizing C build-up and reducing background
  - also requires the OneNeb inert nebulizer (incl. with the Organics kit)
- To enable low ppb level detection of As, Se or Hg, use the Multimode Sample Introduction System (MSIS)
  - also requires the 5 channel peristaltic pump option





# Organics Kit – Inert OneNeb Nebulizer

- Robust PFA and PEEK construction
  - Inert - resistant to strong acids such as HF
  - Resistant to breakage
- Moulded plastic design provides improved nebulizer to nebulizer reproducibility
- Constant diameter narrow bore tubing through to nebulizer tip
  - Ideal for high solids/particulates
  - Improved tolerance to high TDS samples
- Narrow aerosol size distribution provides improved precision
- Compatible with a wide flow range from 0.1 to 2 mL/min.
  - No sensitivity loss at low flow rates



# Principle of Operation – Inert OneNeb Nebulizer

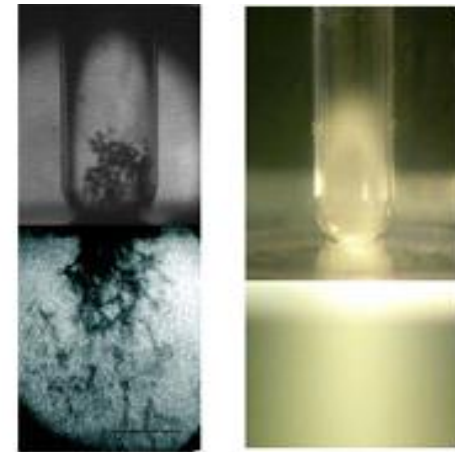
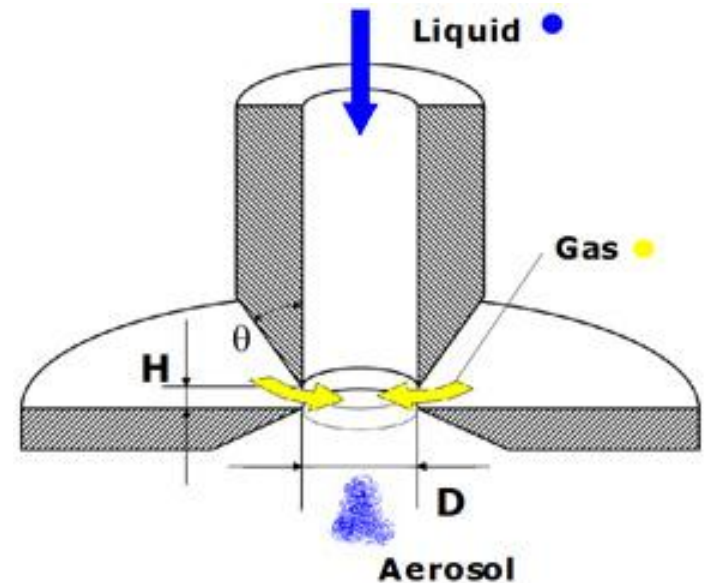
Tip geometry dimensioned to allow carrier gas to mix with the sample

Turbulent mixing of liquid and gas occurs

- Produces aerosol with a narrow size distribution range

Droplets mixed into gas flow

No area of low pressure



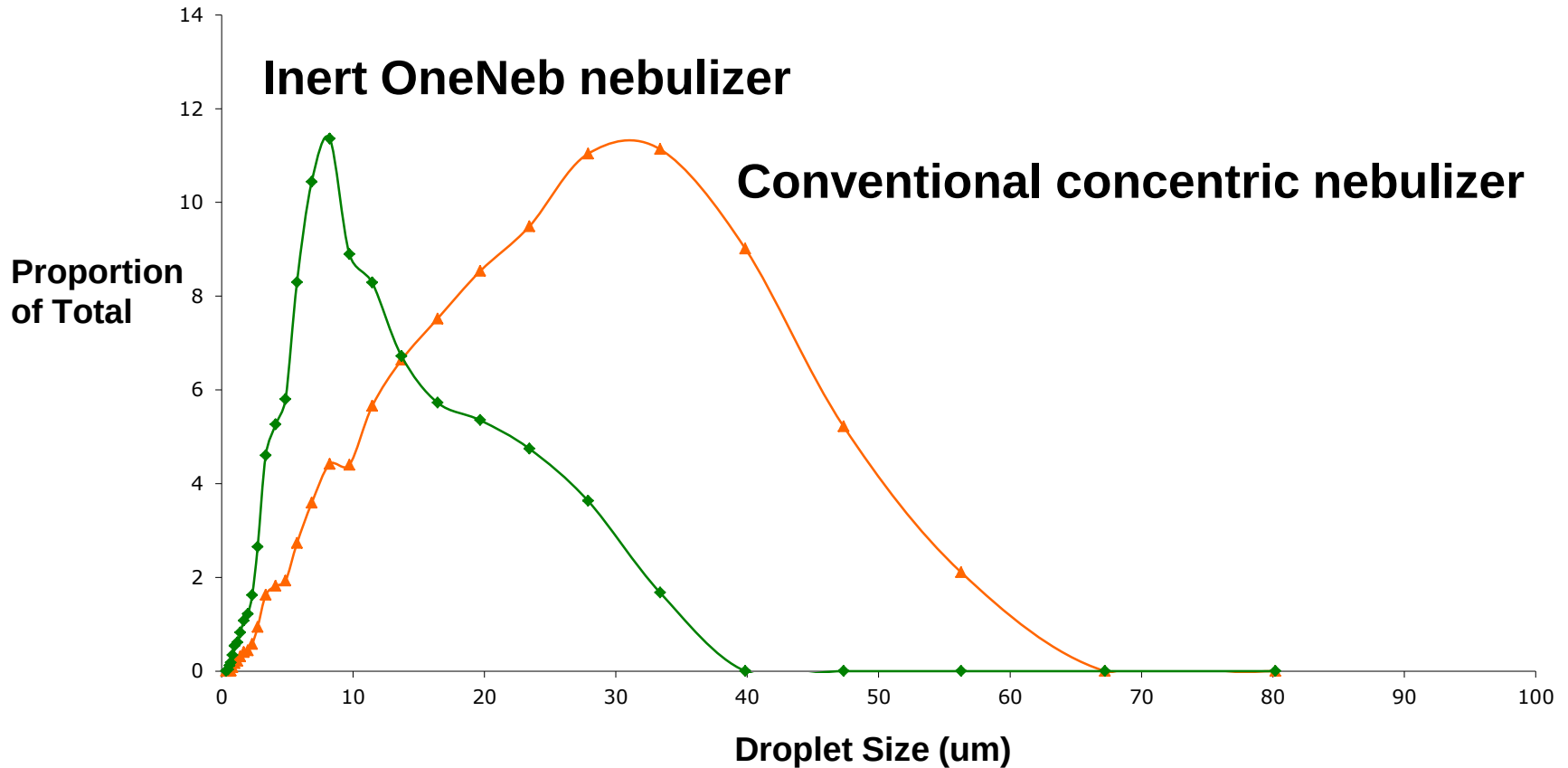
(a)

(b)

# Comparison of Droplet Size Distribution

**Unique Flow Blurring action increases nebulization efficiency**

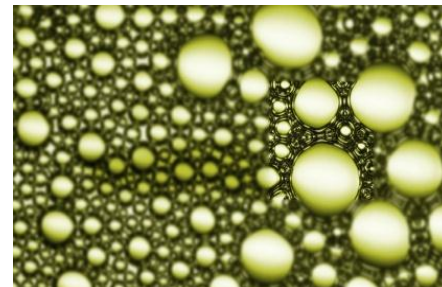
- 3x greater efficiency than conventional concentric nebulizer
- Improved sensitivity



# Applications for the Agilent 4100 MP-AES



**Geochemistry, Mining  
and Minerals**



**Chemical and Petrochemical**



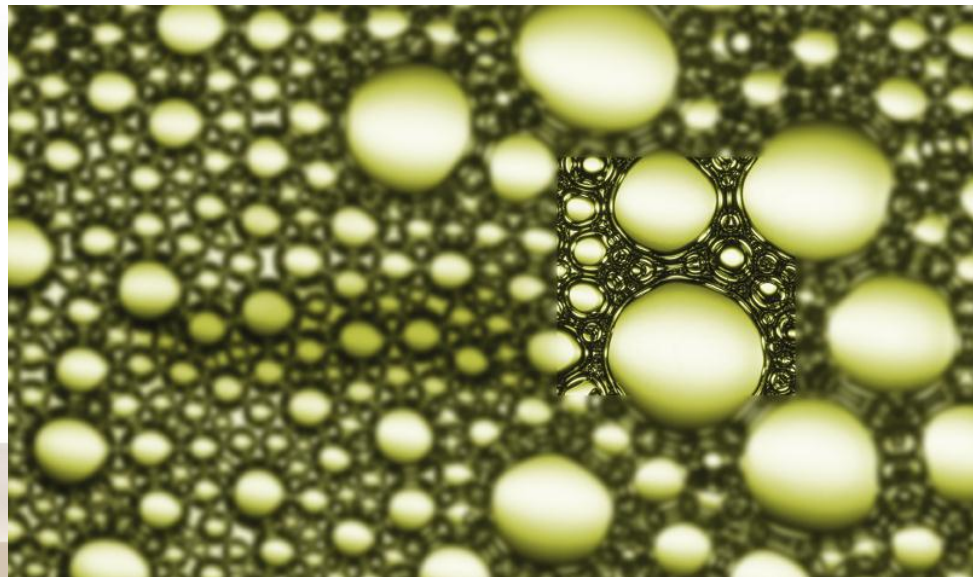
**Food and Agriculture**



**Environmental**

# Applications for the Agilent 4100 MP-AES

Chemical and Petrochemical  
Applications





# Applications MP-AES Can Do – by Market

## GEOCHEMICAL

Geochem samples in aqua regia digests  
Trace elements in geological samples  
Trace level gold in cyanide leach  
Analysis of high purity gold  
Platinum group elements in ore grade material  
Various elements in plating solutions

## CHEMICAL & PETROCHEMICAL

Additives in lubricating oils  
Wear metal contaminants in used oils  
Analysis of coolant  
Analysis of petroleum and diesel fuel  
Major elements in polymers

## FOOD & AGRICULTURE

Major elements in foods, beverages and agricultural samples  
Cations in soils  
Nutrients in soils  
Metals in soil extracts  
Metals in agricultural soil samples

## ENVIRONMENTAL

Hg, Pb, Cd and Cr in electronics and plastics (for WEEE/RoHs compliance)  
Heavy metals in soils  
As, Sb and Se in sediments and waste  
Analysis of waste waters, sediments and soils



# Organic Applications – The Challenge

High vapour  
pressure from  
solvents

- Plasma instability
- May extinguish the plasma

Carbon build up in  
injector

- Poor precision and drift
- Down time - injector may require regular cleaning

Nebulizer  
blockage

- Poor precision
- Down time - nebulizer needs cleaning





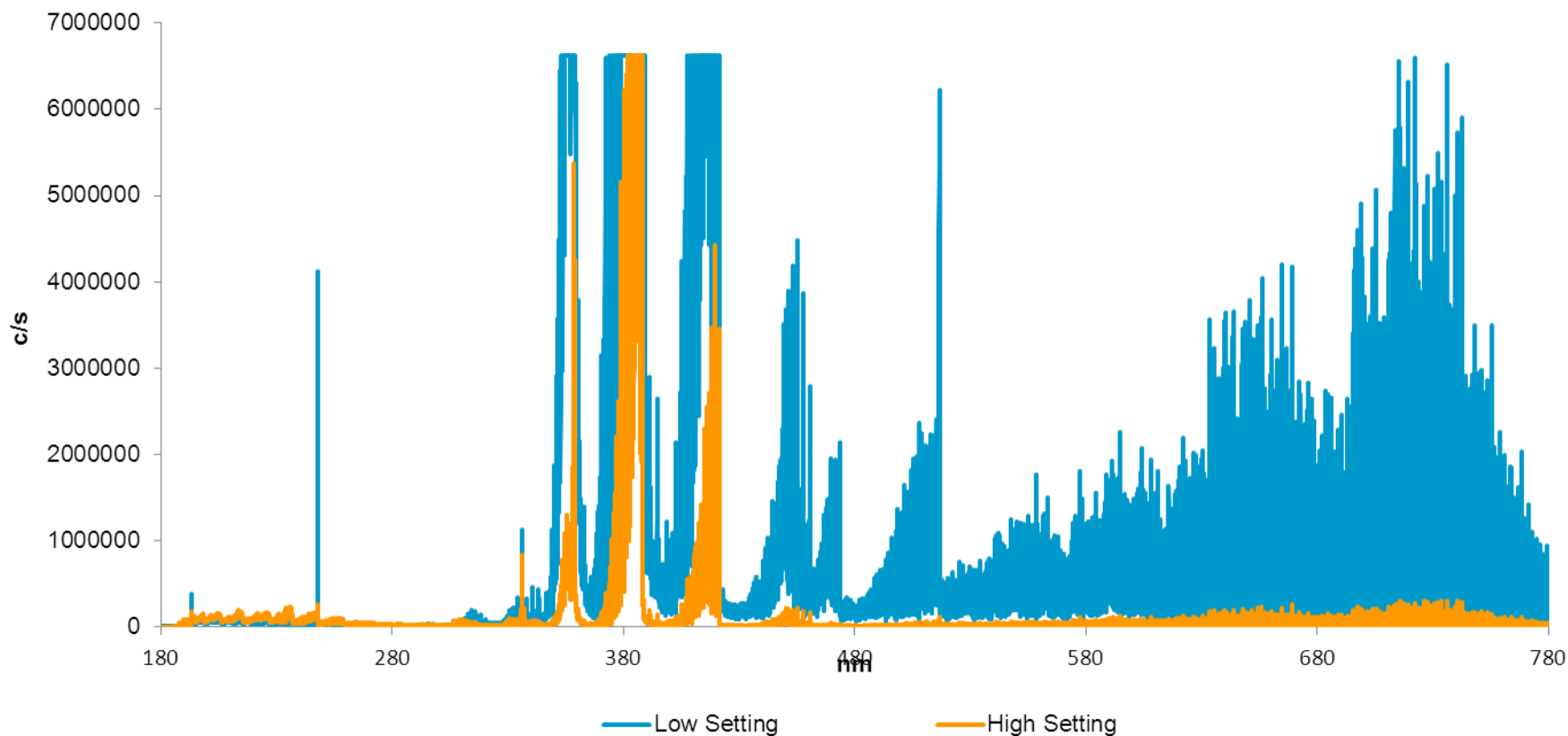
# Organics Kit - External Gas Control Module (EGCM)



1. Prevents carbon build up on the injector when running organics
2. Reduces the background emissions from the plasma
3. A controlled flow of air is bled into the Auxiliary gas flow through the torch

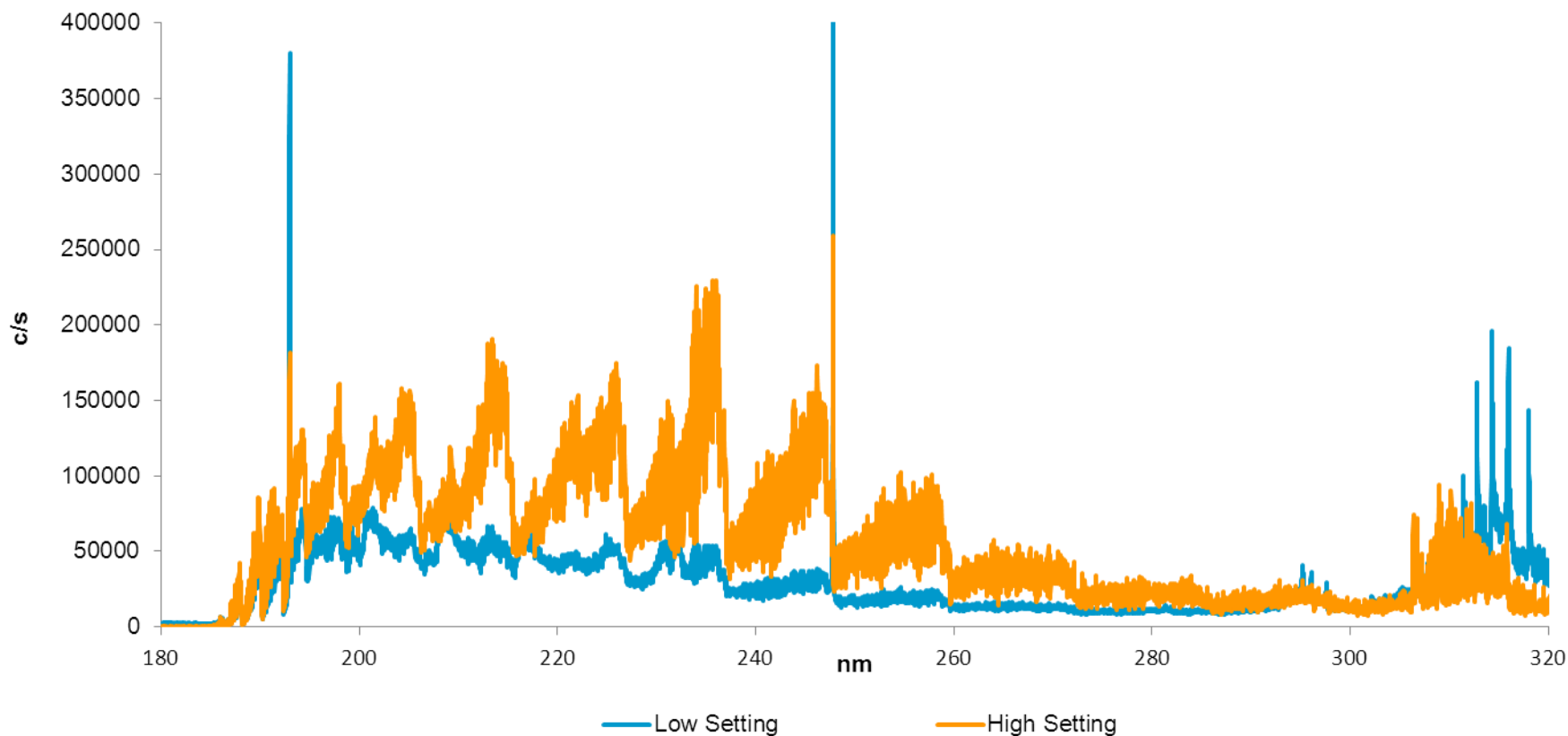
# Solvent Background can be Minimized with EGCM

Shellsol T Background



# Solvent Background can be Minimized with EGCM

Shellsol T Background



# Wear Metals Analysis and Additives in Lubricating Oils



# Importance of Wear Metal Trend Analysis

| Element | Indicator                                |
|---------|------------------------------------------|
| Si      | Dirt intrusion from improper air cleaner |
| Fe      | Wear from engine block, cylinder, etc    |
| Cu      | Wear in piston pins, valve guides, etc   |
| Ni      | Wear gears & certain bearings, etc       |
| Cr      | Piston, rings wear                       |
| Na      | Indicates anti-freeze leakage            |
| Zn      | Antiwear additive in oils                |
| P       | Antiwear additive in oils                |
| Ba      | Used in detergent, oils, rust inhibitors |
| Ca      | Detergents and dispersants               |

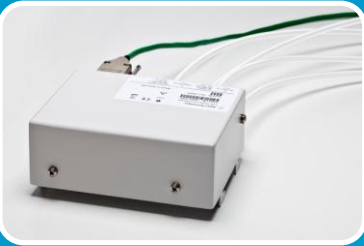


# Challenges with Wear Metal Determinations



## Reduce plasma solvent loading

- OneNeb nebulizer + Double Pass spray chamber
- Orange/Green solvent resistant tubing
- 5 rpm



## Control the background

- Medium setting for Phosphorus at 213.618 nm
- High setting for all others



## Improve Throughput and Ease of use

- SPS 3 with fast pump during sample uptake and rinse
- Auto-optimization of nebulizer pressure and viewing position
- Auto background correction



# Standard and Sample Preparation

Wear metals and contaminants

- 1:10 dilution with Shellsol

Additives

- 1:100 dilution with Shellsol

Standards

- Prepared from 500 ppm S21 (Conostan)

All matrix matched with blank oil for constant viscosity



# Instrument Parameters

| Parameter             | Setting                        |
|-----------------------|--------------------------------|
| Nebulizer             | Inert OneNeb                   |
| Spray chamber         | Double-pass glass cyclonic     |
| Sample tubing         | Orange/green solvent resistant |
| Waste tubing          | Blue/blue solvent resistant    |
| Read Time             | 3 s                            |
| Replicates            | 3                              |
| Stabilization time    | 15 s                           |
| Rinse time            | 45 s                           |
| Fast Pump (80 rpm)    | On                             |
| Background correction | Auto                           |
| Pump speed            | 5 rpm                          |



# Measured Results for SRM 1085b Wear Metals

| Element & wavelength (nm) | Measured (mg/kg) | Certified (mg/kg) | Recovery (%) |
|---------------------------|------------------|-------------------|--------------|
| Fe 259.940                | 314.7 ± 0.3      | 301.2 ± 5.0       | 104          |
| Mn 259.372                | 289.9 ± 0.2      | 300.7 ± 2.0       | 96           |
| Cd 226.502                | 290.9 ± 2.9      | 302.9 ± 5.1       | 96           |
| Cr 276.653                | 305.2 ± 0.1      | 302.9 ± 3.9       | 101          |
| Si 251.611                | 295.7 ± 1.9      | 300.2 ± 5.0       | 99           |
| Ni 305.081                | 291.6 ± 0.1      | 295.9 ± 7.4       | 99           |
| Cu 327.395                | 300.9 ± 0.1      | 295.6 ± 8.5       | 102          |
| Ag 328.068                | 308 ± 0.2        | 304.6 ± 8.9       | 101          |
| Pb 283.305                | 296.1 ± 0.1      | 297.7 ± 6.8       | 99           |
| V 310.229                 | 287.6 ± 0.1      | 297.8 ± 4.6       | 97           |
| Ti 323.452                | 293.9 ± 0.1      | 301.1 ± 2.9       | 98           |
| Sn 303.411                | 295.3 ± 0.3      | 299.4 ± 4.8       | 99           |
| Mo 319.398                | 296.9 ± 0.1      | 300.6 ± 3.2       | 99           |
| Al 396.152                | 291.7 ± 0.2      | 300.4 ± 9.3       | 97           |
| Na 589.592                | 297.4 ± 0.1      | 305.2 ± 7.0       | 97           |

Great recoveries for all key elements



# Measured Recoveries on 10 ppm Spike of Gear Oil

| Element | Wavelength (nm) | Unspiked gear oil (ppm) | Spiked gear oil (ppm) | Spike recovery (%) |
|---------|-----------------|-------------------------|-----------------------|--------------------|
| Ag      | 328.068 nm      | 0.27                    | 11.01                 | 105                |
| Al      | 396.152 nm      | 0.32                    | 10.31                 | 98                 |
| Cd      | 228.802 nm      | 0.14                    | 9.85                  | 95                 |
| Cr      | 276.653 nm      | 0.25                    | 9.92                  | 95                 |
| Cu      | 327.395 nm      | 2.68                    | 13.14                 | 103                |
| Fe      | 259.940 nm      | 10.41                   | 20.09                 | 95                 |
| Mn      | 259.372 nm      | 0.80                    | 11.54                 | 105                |
| Mo      | 319.398 nm      | 9.02                    | 19.34                 | 101                |
| Na      | 589.592 nm      | 0.46                    | 10.70                 | 100                |
| Ni      | 305.081 nm      | 0.07                    | 10.13                 | 99                 |
| Pb      | 283.305 nm      | 0.25                    | 11.36                 | 109                |
| Si      | 251.611 nm      | 2.23                    | 11.60                 | 92                 |
| Sn      | 303.411 nm      | 0.16                    | 10.62                 | 103                |
| Ti      | 323.452 nm      | 0.01                    | 10.87                 | 106                |
| V       | 310.229 nm      | 0.15                    | 10.71                 | 104                |

Great recoveries  
for all key  
elements



# Measured Results for Additive Elements

## Measured Results vs Certified Values

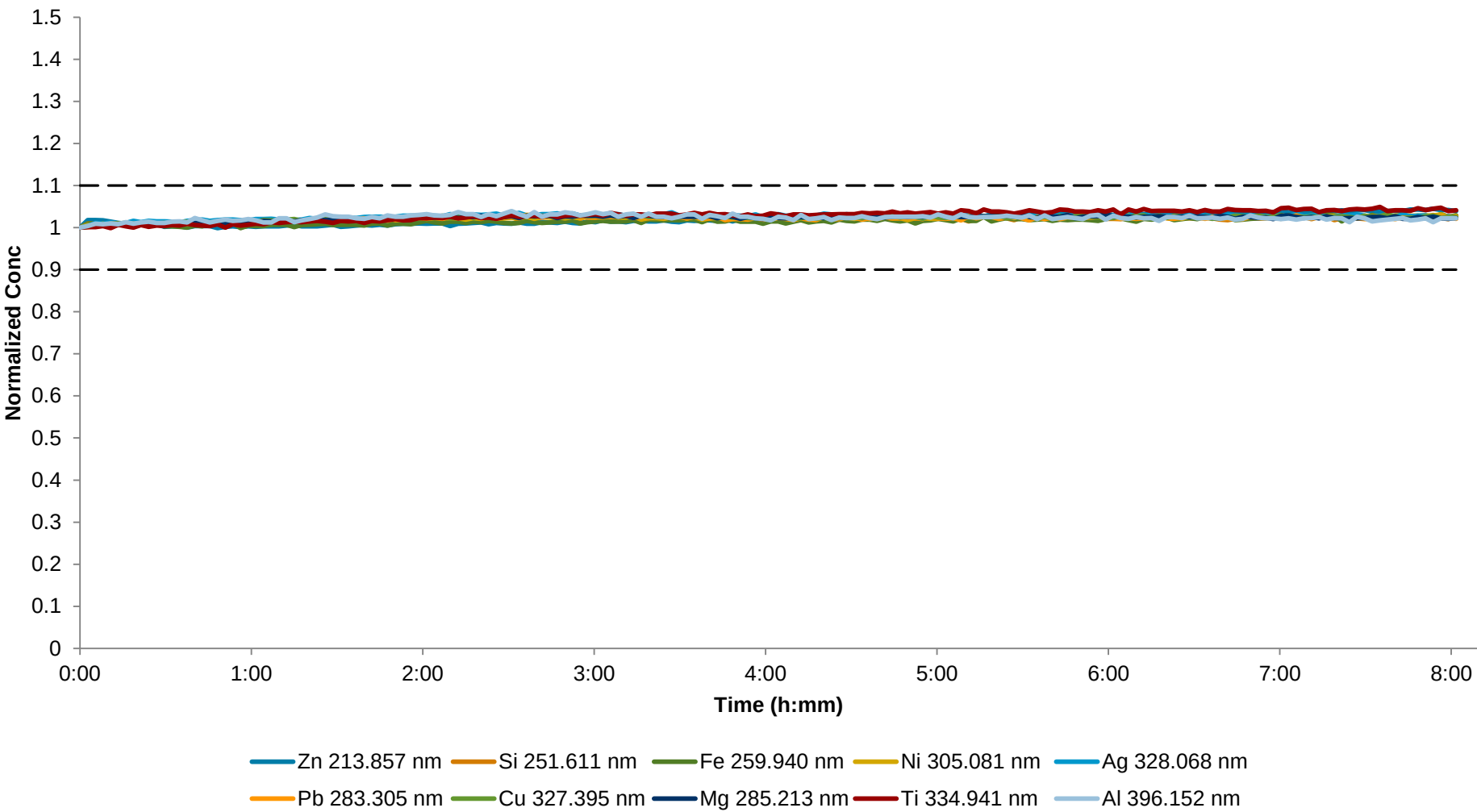
| Element    | Measured (mg/kg) | Certified (mg/kg) | Recovery % |
|------------|------------------|-------------------|------------|
| P 213.618  | 301.5 ± 0.1      | 299.9 ± 7.2       | 101        |
| Zn 481.053 | 314.9 ± 0.3      | 296.8 ± 6.8       | 106        |
| Mg 285.213 | 300.6 ± 0.2      | 297.3 ± 4.1       | 101        |
| Ca 422.673 | 279.6 ± 0.1      | 298               | 94         |
| Ba 614.171 | 281.2 ± 0.1      | 300.1 ± 2.4       | 94         |

## Measured Recoveries on 10 ppm Spike of Gear Oil

| Element    | Mixed Gear (ppm) | Spike (ppm) | Recovery % |
|------------|------------------|-------------|------------|
| P 213.618  | 17.16            | 26.71       | 95         |
| Zn 481.053 | 6.99             | 17.17       | 101        |
| Mg 285.213 | 1.53             | 11.32       | 97         |
| Ca 422.673 | 8.89             | 19.69       | 107        |
| Ba 614.171 | 0.00             | 9.16        | 91         |



# Organics – Long Term Stability



# Determination of Impurities in Gasoline and Petro-Diesel Fuels





# Limits on Target Elements in Diesel Fuel - EN14538



| Property                                           | ASTM Method     | Limits                  | Units                 |
|----------------------------------------------------|-----------------|-------------------------|-----------------------|
| Calcium & Magnesium, combined                      | EN 14538        | 5 max                   | ppm (ug/g)            |
| <b>Flash Point (closed cup)</b>                    | <b>D 93</b>     | <b>93 min.</b>          | <b>Degrees C</b>      |
| Alcohol Control (One of the following must be met) |                 |                         |                       |
| 1. Methanol Content                                | EN14110         | 0.2 Max                 | % volume              |
| 2. Flash Point                                     | D93             | 130 Min                 | Degrees C             |
| <b>Water &amp; Sediment</b>                        | <b>D 2709</b>   | <b>0.05 max.</b>        | <b>% vol.</b>         |
| Kinematic Viscosity, 40 C                          | D 445           | 1.9 - 6.0               | mm <sup>2</sup> /sec. |
| Sulfated Ash                                       | D 874           | 0.02 max.               | % mass                |
| <b>Sulfur</b>                                      |                 |                         |                       |
| <b>S 15 Grade</b>                                  | <b>D 5453</b>   | <b>0.0015 max. (15)</b> | <b>% mass (ppm)</b>   |
| <b>S 500 Grade</b>                                 | <b>D 5453</b>   | <b>0.05 max. (500)</b>  | <b>% mass (ppm)</b>   |
| Copper Strip Corrosion                             | D 130           | No. 3 max.              |                       |
| Cetane                                             | D 613           | 47 min.                 |                       |
| <b>Cloud Point</b>                                 | <b>D 2500</b>   | <b>Report</b>           | <b>Degrees C</b>      |
| Carbon Residue 100% sample                         | D 4530*         | 0.05 max.               | % mass                |
| <b>Acid Number</b>                                 | <b>D 664</b>    | <b>0.50 max.</b>        | <b>mg KOH/g</b>       |
| <b>Free Glycerin</b>                               | <b>D 6584</b>   | <b>0.020 max.</b>       | <b>% mass</b>         |
| <b>Total Glycerin</b>                              | <b>D 6584</b>   | <b>0.240 max.</b>       | <b>% mass</b>         |
| Phosphorus Content                                 | D 4951          | 0.001 max.              | % mass                |
| Distillation, T90 AET                              | D 1160          | 360 max.                | Degrees C             |
| Sodium/Potassium, combined                         | EN 14538        | 5 max                   | ppm                   |
| <b>Oxidation Stability</b>                         | <b>EN 14112</b> | <b>3 min</b>            | <b>hours</b>          |



**Workmanship** Free of undissolved water, sediment, & suspended matter  
**BOLD = 8Q-9000 Critical Specification Testing Once Production Process Under Control**

\* The carbon residue shall be run on the 100% sample.



# Sample Preparation – Method EN14538

A commercial diesel sample was analysed

- 1:10 dilution with Shellsol

Standards made from Conostan S21+K

- 0.5 ppm, 1 ppm, 5 ppm, 10 ppm

All samples and standards matrix matched  
with blank oil



# Instrument Parameters

| Parameter             | Setting                        |
|-----------------------|--------------------------------|
| Nebulizer             | Inert OneNeb                   |
| Spray chamber         | Double-pass glass cyclonic     |
| Sample tubing         | Orange/green solvent resistant |
| Waste tubing          | Blue/blue solvent resistant    |
| Read Time             | 3 s                            |
| Replicates            | 3                              |
| Stabilization time    | 15 s                           |
| Fast Pump (80 rpm)    | On                             |
| Background correction | Auto                           |
| Pump speed            | 5 rpm                          |



# Challenges with Petro-Diesel Fuel Determinations



## Reduce plasma solvent loading

- OneNeb nebulizer + Double Pass spray chamber
- Orange/Green solvent resistant tubing
- 5 rpm



## Control the background

- High setting used for all elements



## Improve Throughput and Ease of use

- SPS 3 with fast pump during sample uptake and rinse
- Auto-optimization of nebulizer pressure and viewing position
- Auto background correction

# Method Detection Limits

$3\sigma$ , 3s read time, 10 blanks

| Element | Wavelength (nm) | MDL (ppb) |
|---------|-----------------|-----------|
| Mg      | 285.213         | 2.7       |
| Ca      | 422.673         | 8.2       |
| Na      | 588.995         | 18.7      |
| K       | 766.491         | 2.7       |



# Spike Recoveries on Diesel Fuel

Diesel fuel sample spiked with 0.55 ppm S21+K

| Element       | Sample (ppm) | Spiked Sample (ppm) | Recovery (%) |
|---------------|--------------|---------------------|--------------|
| Mg 285.213 nm | < MDL        | 0.53                | 97           |
| Ca 422.673 nm | < MDL        | 0.51                | 93           |
| Na 588.995 nm | < MDL        | 0.51                | 93           |
| K 766.491 nm  | < MDL        | 0.51                | 93           |

# Direct Measurement of Impurities in Gasoline

Same ASTM D6751 limits

- Ca, Mg combined 5 ppm
- Na, K combined 5 ppm

High volatility of the gasoline is a challenge

- Cooled spray chamber -10 °C
- Standard Additions calibration
- Direct measurement of samples





# Instrument Parameters

| Parameter             | Setting                               |
|-----------------------|---------------------------------------|
| Nebulizer             | Inert OneNeb                          |
| Spray chamber         | IsoMist cooled spray chamber (-10 °C) |
| Sample tubing         | Orange/green solvent resistant        |
| Waste tubing          | Blue/blue solvent resistant           |
| Read Time             | 3 s                                   |
| Replicates            | 3                                     |
| Stabilization time    | 30 s                                  |
| Fast Pump (80 rpm)    | On                                    |
| Background correction | Off-peak                              |
| Pump speed            | 5 rpm                                 |



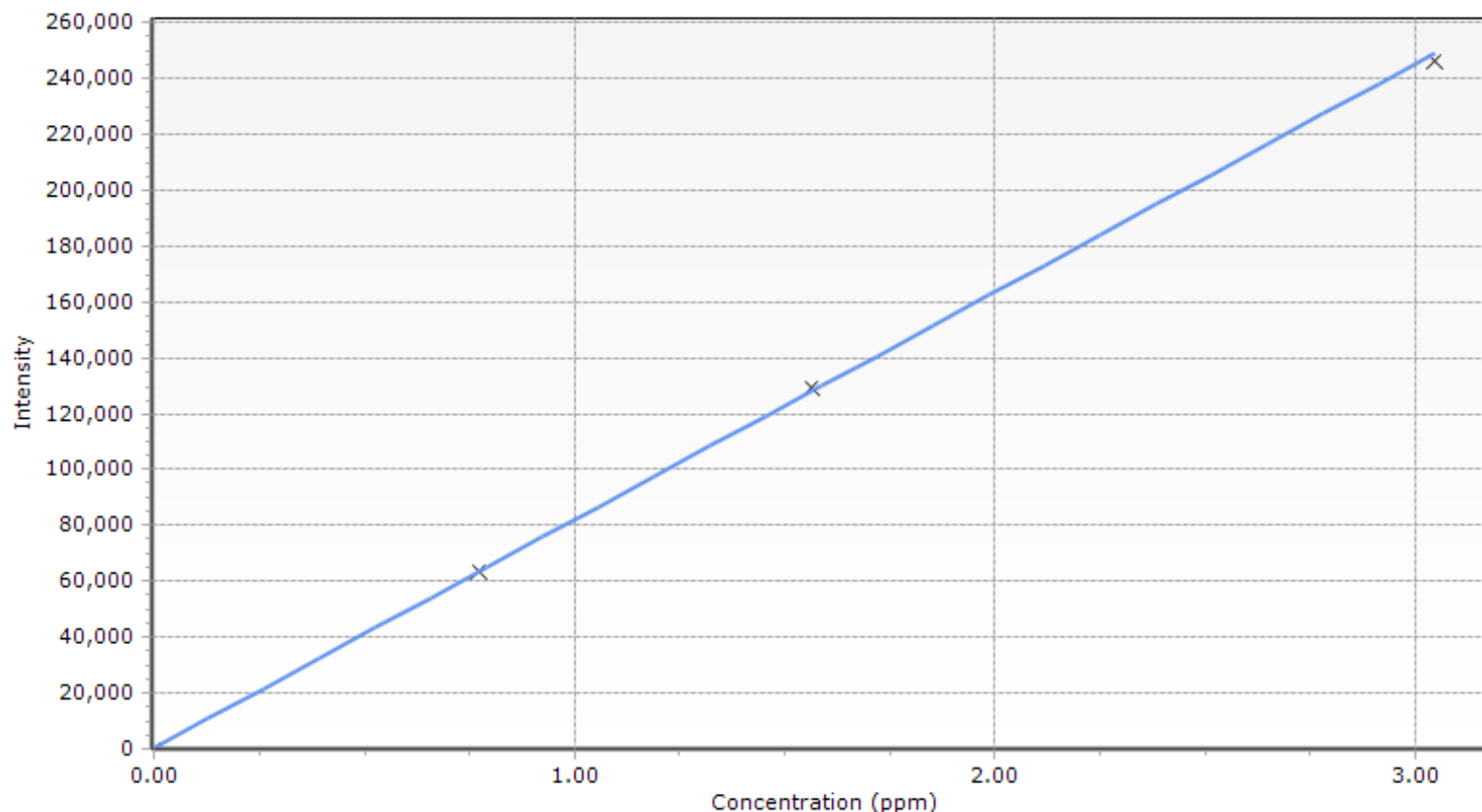
# Calibrations and Detection Limits

| Element | Wavelength (nm) | Correlation Coeff | MDL (ppb) |
|---------|-----------------|-------------------|-----------|
| Mg      | 285.213         | 0.99993           | 2.7       |
| Ca      | 422.673         | 0.99934           | 4.3       |
| Na      | 588.995         | 0.99939           | 5.3       |
| K       | 766.491         | 0.99975           | 29.4      |



# Excellent Linearity with Standard Additions - Gasoline

Mg 285.213 nm



Intensity = 81779.569 \* Concentration + 239.829  
Correlation coefficient: 0.99993

# Spike Recoveries on Gasoline

Gasoline fuel sample spiked with 1.1 ppm S21+K

| Element       | Sample (ppm) | Spike (ppm) | Recovery % |
|---------------|--------------|-------------|------------|
| Mg 285.213 nm | < MDL        | 1.11        | 100        |
| Ca 422.673 nm | < MDL        | 1.06        | 95         |
| Na 588.995 nm | < MDL        | 1.11        | 100        |
| K 766.491 nm  | 0.05         | 1.12        | 96         |

# 4100 MP-AES Solves the Challenges



High vapour  
pressure from  
solvents

- OneNeb nebulizer
- Reduced sample flow
- Cooled spray chamber

Carbon build up in  
injector

EGCM

Nebulizer  
blockage

- OneNeb nebulizer



# Applications MP-AES Can Do – by Market

## GEOCHEMICAL

Geochem samples in aqua regia digests  
Trace elements in geological samples  
Trace level gold in cyanide leach  
Analysis of high purity gold  
Platinum group elements in ore grade material  
Various elements in plating solutions

## CHEMICAL & PETROCHEMICAL

Additives in lubricating oils  
Wear metal contaminants in used oils  
Analysis of coolant  
Analysis of petroleum and diesel fuel  
Major elements in polymers

## FOOD & AGRICULTURE

Major elements in foods, beverages and agricultural samples  
Cations in soils  
Nutrients in soils  
Metals in soil extracts  
Metals in agricultural soil samples

## ENVIRONMENTAL

Hg, Pb, Cd and Cr in electronics and plastics (for WEEE/RoHs compliance)  
Heavy metals in soils  
As, Sb and Se in sediments and waste  
Analysis of waste waters, sediments and soils



# Applications for the Agilent 4100 MP-AES

Geochemical, Metals and  
Mining Applications



# Mining (Geochemistry) Applications

Aqua regia digests, fire assay, cyanide leaches

- Au, Ag, Pt, Pd, Cu, Ni, Zn

## Analytical Challenges

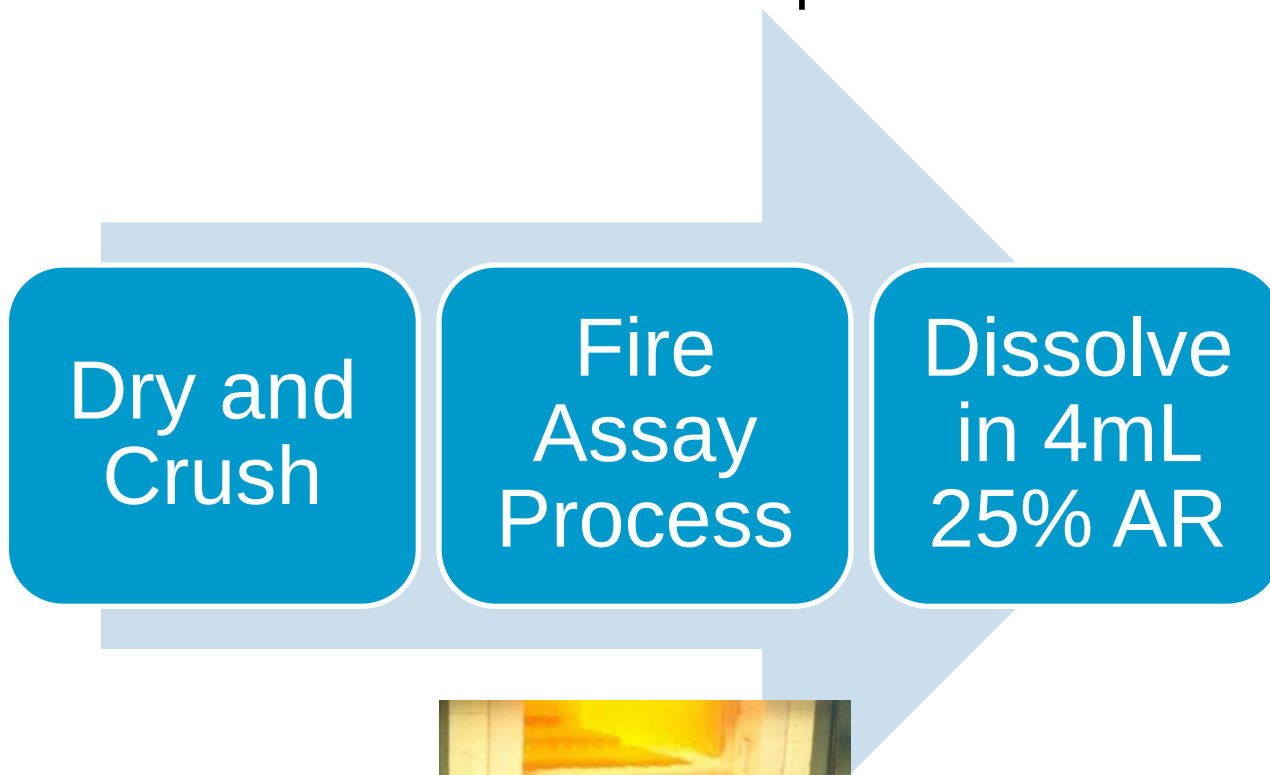
- Low detection limits
- High levels of dissolved solids
- Linearity
- Sample throughput





# Gold and Precious Metals by Fire Assay

Au, Pt and Pb extracted into a silver sphere

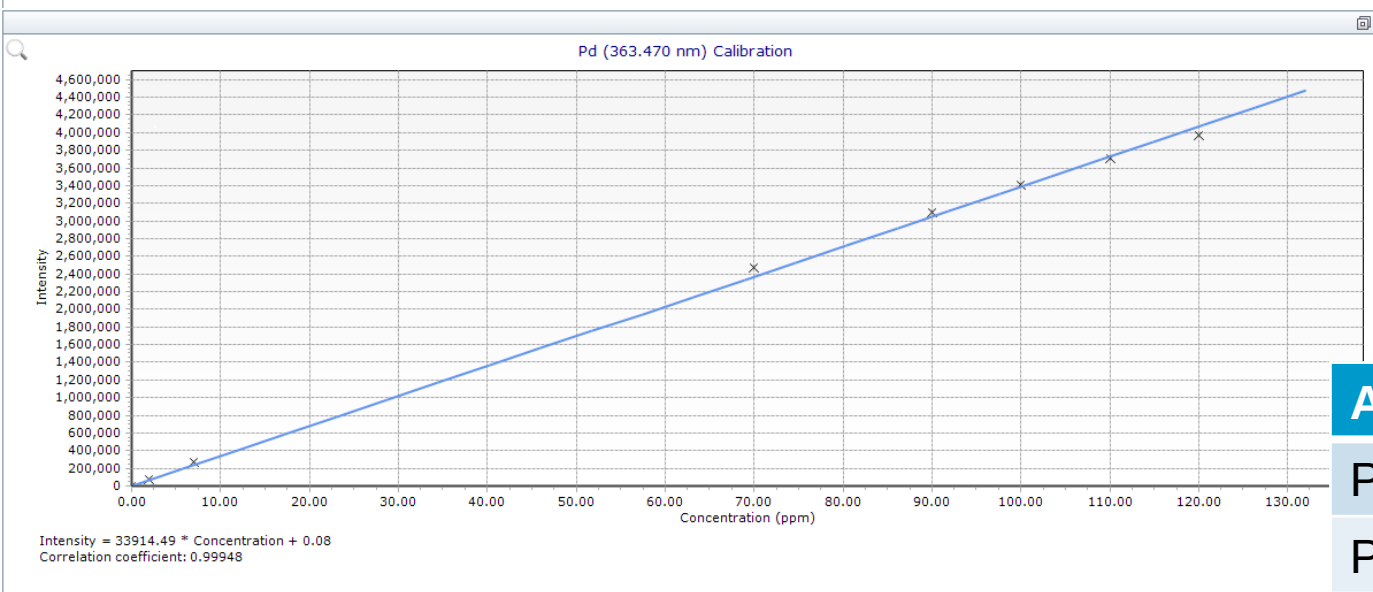
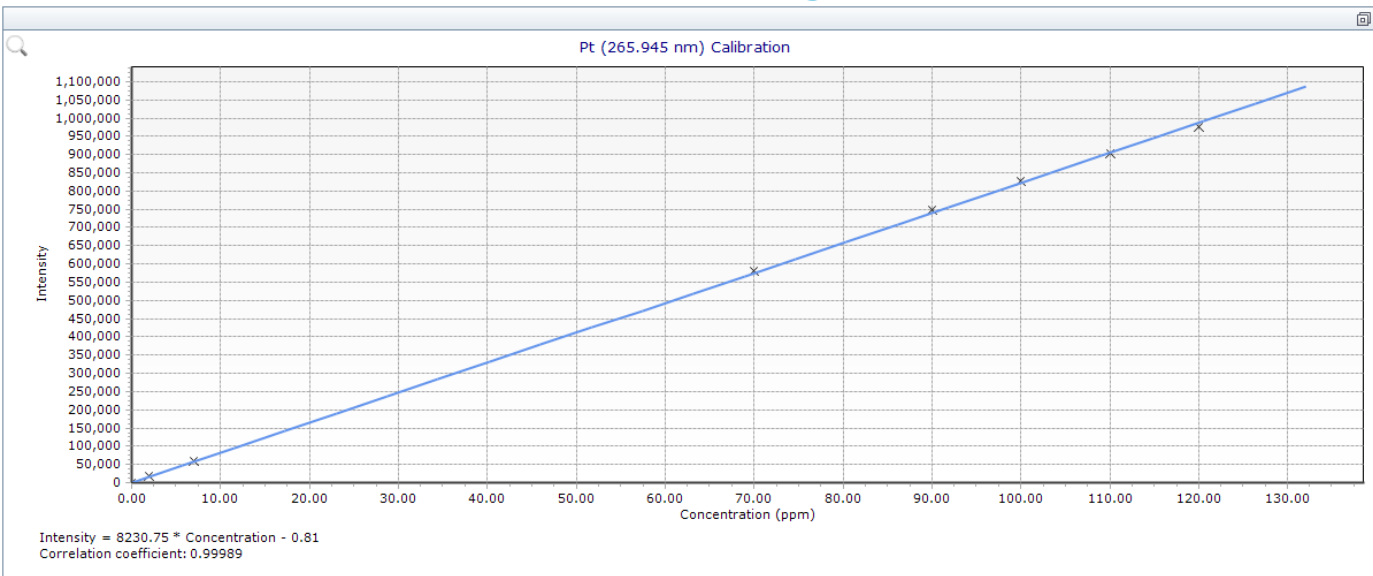


# Instrument Parameters

| Parameter             | Setting                         |
|-----------------------|---------------------------------|
| Nebulizer             | Glass concentric                |
| Spray chamber         | Single-pass glass cyclonic      |
| Read Time             | 3 s                             |
| Replicates            | 3                               |
| Sample uptake delay   | 10 s                            |
| Stabilization time    | 5 s                             |
| Fast Pump             | On (80 rpm)                     |
| Background correction | Auto                            |
| Nebulizer Pressure    | 140 to 240 kPa (auto optimized) |



# Extended Linear Range for Platinum & Palladium



**Both Pt & Pd  
linear to  
120 ppm**

| Analyte    | Correlation |
|------------|-------------|
| Pt 265.495 | 0.9999      |
| Pd 363.470 | 0.9995      |

# Accuracy –Platinum & Palladium in Custom CRMs

## Platinum

| CRM | Certified value (mg/L) | MP-AES result (mg/L) |
|-----|------------------------|----------------------|
| 6   | 0.74                   | 0.75                 |
| 7   | 35.6                   | 35.9                 |
| 8   | 9.0                    | 9.5                  |

## Palladium

| CRM | Certified value (mg/L) | MP-AES result (mg/L) |
|-----|------------------------|----------------------|
| 6   | 3.21                   | 3.4                  |
| 7   | 44.4                   | 44.0                 |
| 8   | 35.0                   | 36.5                 |



# Superior Performance – Detection Limits

| Analyte | Wavelength (nm) | Method DL (ug/L) | Flame AA IDL (ug/L) |
|---------|-----------------|------------------|---------------------|
| Au      | 267.595         | 4                | 11                  |
| Pt      | 265.945         | 13               | 76                  |
| Pd      | 363.469         | 0.7              | 15                  |

**MDL =  $3\sigma$ , 3s read time, 20 blanks**



# Customer Data

## Analysis of Rocklabs Certified Reference Material OxH97

### **Origin of Reference Material:**

Basalt and feldspar minerals with minor quantities of finely divided gold-containing minerals that have been screened to ensure there is no gold nugget effect.

**Data generated at Swastika Labs Ontario, Canada**

# ANALYTICAL CONDITIONS

Samples were run over a period of 6 months as QC insertions in routine analytical runs.

Typically samples run in positions 12, 32, and 54 of autosampler batches with NO recalibration throughout the run.

Front end setup:

- Seaspray nebulizer

- Single pass spray chamber

- Standard MP torch

- Pump tubing – orange green

## Data and Results

| Sequence                                   | Sample Batch No | Results (g/MT) |
|--------------------------------------------|-----------------|----------------|
| 1                                          | 12-1452 -1      | 1.29           |
| 2                                          | 12-1452 -2      | 1.27           |
| 3                                          | 12-1453 -1      | 1.26           |
| 4                                          | 12-1453 -2      | 1.26           |
| 5                                          | 12-1453 -3      | 1.27           |
| 6                                          | 12-2307 -1      | 1.25           |
| Average of 100 data points over six months |                 | 1.28 g/MT      |
| Calculated Standard Deviation              |                 | 0.02           |
| Certified Value (RockLabs)                 |                 | 1.278+/-0.014  |



# MP-AES Improves Throughput over Flame AA

Ore analysis can be volume limited

Typical sample volume available ~4mL

Automate analysis & reduce sample consumption using the autosampler

- Analysis time only 55s sample to sample
- Sample consumption ~1.8 mL
- Throughput 65 samples/hour, or 520 samples in 8 hours
- Plus...
- Safe unattended, multielement operation



**Agilent SPS 3 Autosampler**

# Applications for the Agilent 4100 MP-AES

Food and Agricultural  
Applications



# Applications the MP-AES can do – by Market

## GEOCHEMICAL

Geochem samples in aqua regia digests  
Trace elements in geological samples  
Trace level gold in cyanide leach  
Analysis of high purity gold  
Platinum group elements in ore grade material  
Various elements in plating solutions

## CHEMICAL & PETROCHEMICAL

Additives in lubricating oils  
Wear metal contaminants in used oils  
Analysis of coolant  
Analysis of petroleum and diesel fuel  
Major elements in polymers

## FOOD & AGRICULTURE

Major elements in foods, beverages and agricultural samples  
Cations in soils  
Nutrients in soils  
Metals in soil extracts  
Metals in agricultural soil samples

## ENVIRONMENTAL

Hg, Pb, Cd and Cr in electronics and plastics (for WEEE/RoHs compliance)  
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As, Sb and Se in sediments and waste  
Analysis of waste waters, sediments and soils

# Food Applications – Types of Measurement



## Product Labeling

- Nutrient Levels



## Food Quality

- Contamination
- Pollutants



## Food Production

- QA/QC



## Research

- Developing Better Products



# Which Elements are Monitored in Foods?

## Toxic trace elements

- Low level analysis – typically ppb levels
- As, Cd, Hg, Pb, Tl, Cr

## Common elements that are toxic in excess

- Typically range from ppb to ppm level
- Al, Ni, Cu, Zn, Se, Mo, Sn

## Essential minerals

- Can cover a wide range from ppm to % levels
- Na, Mg, P, S, K, Ca, Fe



# Sample Types

## Wide Range of Matrices

- Dairy, Meat, Fish, Grain, Vegetables, Fruit, etc
- Processed goods
  - Canned, frozen, dried and preserved food
  - Drinks (soda, tea, coffee, alcoholic beverages etc.)
  - Preservatives, “fillers” and additives such as calcium carbonate
- “Nutraceuticals”

## Other Important Samples

- Water (used for irrigation)
- Soil
- Fertilizer
- Food additives / flavors / colors
- Packaging material



# Food and Agriculture Applications – The Challenge

## Robust Sample Introduction

- Large number of samples
- Wide range of matrices
- High total dissolved solids (TDS)

## Many Elements Per Sample

- Potential for spectral interferences
- Labs. need high sample throughput
- Labs. also want low cost of analysis

## Wide Dynamic Range

- Trace (ppb) level to % level
- Avoid sample dilution (if possible)



# Major, Minor and Trace Analysis in Foodstuffs



A range of samples

1. NIES CRM No.7 Tea Leaves
2. NIES CRM No.10c Rice Flour
3. NIST SRM 1577 Bovine Liver:

Plus pre-prepared sample solutions in 4 %  $\text{HNO}_3$  (High Purity Standards)

4. CRM-Wheat Flour
5. CRM-Milk Powder
6. CRM-Oyster Tissue



# Instrument Parameters

| Parameter             | Setting                        |
|-----------------------|--------------------------------|
| Nebulizer             | Glass concentric               |
| Spray chamber         | Single pass glass cyclonic     |
| Sample tubing         | White/white                    |
| Waste tubing          | Blue/blue                      |
| Read Time             | 10 s                           |
| Replicates            | 3                              |
| Stabilization time    | 10 s                           |
| Fast Pump             | On (80 rpm)                    |
| Pump speed            | 12 rpm                         |
| Nebulizer pressure    | 160 – 180 kpa (auto optimized) |
| Background Correction | Auto                           |



# Measured Results for CRM Food Samples



## NIES No. 7 Tea Leaves



## High Purity Stds CRM Milk Powder

| Element | Measured values | Certified values |
|---------|-----------------|------------------|
|         | wt%             | wt%              |
| Ca      | 0.314 ± 0.013   | 0.320 ± 0.012    |
| Mg      | 0.150 ± 0.004   | 0.153 ± 0.006    |
| K       | 1.861 ± 0.074   | 1.86 ± 0.07      |
|         | mg/kg           | mg/kg            |
| Ba      | 5.76 ± 0.57     | 5.7*             |
| Cd      | nd              | 0.03 ± 0.03      |
| Co      | nd              | 0.12*            |
| Cr      | nd              | 0.15*            |
| Cu      | 7.13 ± 0.81     | 7 ± 0.3          |
| Pb      | nd              | 0.8 ± 0.03       |
| Ni      | 6.03 ± 0.63     | 6.5 ± 0.3        |
| Sr      | 3.63 ± 0.43     | 3.7*             |
| Zn      | 34 ± 3          | 33 ± 3           |

| Element | Measured values (mg/kg) | Certified values (mg/kg) |
|---------|-------------------------|--------------------------|
| Al      | nd                      | 0.020 ± 0.002            |
| Ca      | 131 ± 9                 | 130 ± 1                  |
| Co      | nd                      | 0.0004*                  |
| Cr      | nd                      | 0.0003*                  |
| Cu      | 0.006 ± 0.001           | 0.007 ± 0.001            |
| Fe      | 0.018 ± 0.002           | 0.020 ± 0.001            |
| K       | 178 ± 6                 | 170 ± 2                  |
| P       | 98.7 ± 1.3              | 100 ± 1                  |
| Pb      | nd                      | 0.002*                   |
| Mg      | 11.9 ± 0.2              | 12 ± 0.1                 |
| Mn      | 0.003 ± 0.002           | 0.003*                   |
| Na      | 48.7 ± 2.6              | 50 ± 1                   |
| Zn      | 0.48 ± 0.05             | 0.50 ± 0.01              |

\* Reference values only



# Measured Results for CRM Food Samples



## NIES No. 10c Rice Flour

| Element | Measured values | Certified values |
|---------|-----------------|------------------|
|         | wt%             | wt%              |
| Mg      | 0.127 ± 0.006   | 0.125 ± 0.008    |
| K       | 0.279 ± 0.012   | 0.275 ± 0.010    |
| P       | 0.300 ± 0.010   | 0.335 ± 0.008    |
|         | mg/kg           | mg/kg            |
| Al      | 1.49 ± 0.13     | 1.5*             |
| Ca      | 95.4 ± 7.0      | 95 ± 2           |
| Cd      | 1.83 ± 0.14     | 1.82 ± 0.06      |
| Co      | nd              | 0.007*           |
| Cr      | nd              | 0.08*            |
| Cu      | 4.03 ± 0.32     | 4.1 ± 0.3        |
| Fe      | 10.6 ± 0.15     | 11.4 ± 0.8       |
| Mo      | nd              | 1.6 ± 0.1        |
| Ni      | nd              | 0.30 ± 0.03      |
| Sr      | 0.2             | 0.2*             |
| Zn      | 21.8 ± 1.0      | 23.1 ± 0.8       |

## High Purity Stds CRM Wheat Flour

| Element | Measured values (mg/kg) | Certified values (mg/kg) |
|---------|-------------------------|--------------------------|
| Al      | 0.83 ± 0.02             | 0.85 ± 0.01              |
| Ca      | 9.64 ± 0.97             | 9.5 ± 0.1                |
| Cd      | nd                      | 0.0015*                  |
| Co      | nd                      | 0.001*                   |
| Cr      | 0.013 ± 0.001           | 0.014*                   |
| Cu      | 0.09 ± 0.008            | 0.1 ± 0.002              |
| Fe      | 0.81 ± 0.04             | 0.90 ± 0.01              |
| K       | 62.5 ± 0.5              | 65 ± 0.7                 |
| P       | 61.1 ± 1.7              | 65 ± 0.7                 |
| Pb      | 0.05 ± 0.001            | 0.050 ± 0.003            |
| Mg      | 20.8 ± 0.1              | 20.0 ± 0.2               |
| Mn      | 0.36 ± 0.02             | 0.4 ± 0.008              |
| Ni      | nd                      | 0.009 ± 0.001            |
| Zn      | 0.47 ± 0.05             | 0.50 ± 0.01              |

\* Reference values only



# Measured Results for CRM Food Samples



## NIST 1577 Bovine Liver

## High Purity Std CRM Oyster Tissue

| Element | Measured values | Certified values |
|---------|-----------------|------------------|
|         | wt%             | wt%              |
| Na      | 0.247 ± 0.006   | 0.243 ± 0.013    |
| K       | 1.00 ± 0.08     | 0.97 ± 0.06      |
|         | mg/kg           | mg/kg            |
| Ca      | 131             | 123*             |
| Cd      | nd              | 0.27 ± 0.04      |
| Co      | nd              | 0.18*            |
| Cu      | 185 ± 6         | 193 ± 10         |
| Fe      | 266 ± 5         | 270 ± 20         |
| Pb      | nd              | 0.34 ± 0.08      |
| Mg      | 625 ± 45        | 605*             |
| Mn      | 10.4 ± 1.41     | 10.3 ± 1         |
| Mo      | nd              | 3.2*             |
| Sr      | 0.15 ± 0.07     | 0.14*            |
| Zn      | 125 ± 4         | 130 ± 10         |

| Element | Measured values (mg/kg) | Certified values (mg/kg) |
|---------|-------------------------|--------------------------|
| Al      | 2.92 ± 0.07             | 3*                       |
| Ca      | 15.0 ± 0.49             | 15*                      |
| Cd      | nd                      | 0.03*                    |
| Co      | nd                      | 0.004*                   |
| Cr      | nd                      | 0.007*                   |
| Cu      | 0.56 ± 0.05             | 0.6*                     |
| K       | 100 ± 0.96              | 100*                     |
| P       | 79.1 ± 0.9              | 80*                      |
| Pb      | nd                      | 0.005*                   |
| Mg      | 12.1 ± 0.2              | 12*                      |
| Mn      | 0.18 ± 0.01             | 0.2*                     |
| Na      | 48.9 ± 0.8              | 50*                      |
| Ni      | nd                      | 0.01*                    |
| Zn      | 8.3 ± 0.4               | 9*                       |

\* Reference values only



# Applications the MP-AES can do – by Market

## GEOCHEMICAL

Geochem samples in aqua regia digests  
Trace elements in geological samples  
Trace level gold in cyanide leach  
Analysis of high purity gold  
Platinum group elements in ore grade material  
Various elements in plating solutions

## CHEMICAL & PETROCHEMICAL

Additives in lubricating oils  
Wear metal contaminants in used oils  
Analysis of coolant  
Analysis of petroleum and diesel fuel  
Major elements in polymers

## FOOD & AGRICULTURE

Major elements in foods, beverages and agricultural samples  
Cations in soils  
Nutrients in soils  
Metals in soil extracts  
Metals in agricultural soil samples

## ENVIRONMENTAL

Hg, Pb, Cd and Cr in electronics and plastics (for WEEE/RoHs compliance)  
Heavy metals in soils  
As, Sb and Se in sediments and waste  
Analysis of waste waters, sediments and soils



# Instrument Parameters

| Parameter          | Setting                          |
|--------------------|----------------------------------|
| Nebulizer          | Glass concentric                 |
| Spray chamber      | Single pass glass cyclonic       |
| Sample tubing      | Black/black                      |
| Waste tubing       | Blue/blue                        |
| Read Time          | 3 s                              |
| Replicates         | 3                                |
| Stabilization time | 10 s                             |
| Fast Pump (80 rpm) | On                               |
| Pump speed         | 15 rpm                           |
| Viewing position   | -20 to 20 steps (auto optimized) |
| Nebulizer pressure | 120 – 240 kpa (auto optimized)   |



# Measured Results for NIST SRM 2710 Montana Soil

| Analyte | MP-AES Results<br>Average (mg/kg) | NIST Reference<br>Range (mg/kg) |
|---------|-----------------------------------|---------------------------------|
| Al      | 24300 ± 400                       | 12000 - 26000                   |
| As      | 550 ± 20                          | 490 - 600                       |
| Cr      | 21 ± 1                            | 15 - 23                         |
| Cu      | 2800 ± 20                         | 2400 - 3400                     |
| Fe      | 28000 ± 300                       | 22000 - 32000                   |
| Mn      | 8500 ± 200                        | 6200 - 9000                     |
| Ni      | 9.5 ± 0.6                         | 8.8 - 15                        |
| Pb      | 5600 ± 300                        | 4300 - 7000                     |
| Zn      | 6100 ± 200                        | 5200 - 6900                     |

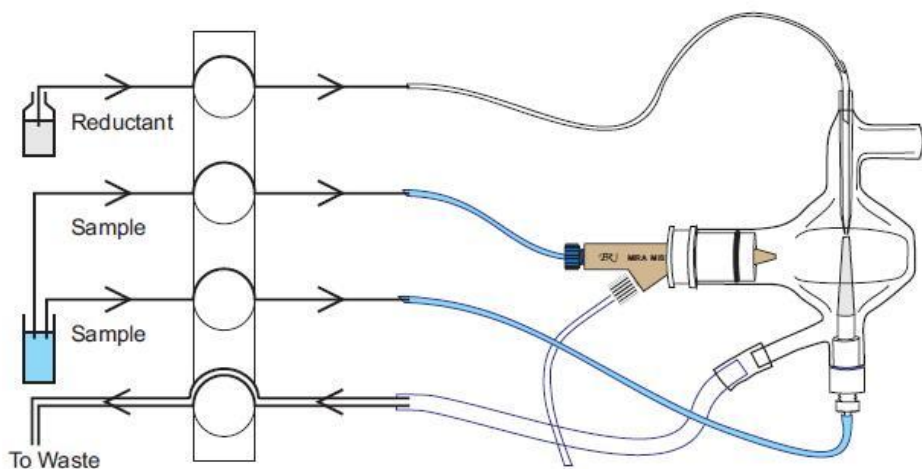
# Measured Results for NIST SRM 2711 Montana Soil

| Analyte | MP-AES Results<br>Average (mg/kg) | NIST Reference<br>Range (mg/kg) |
|---------|-----------------------------------|---------------------------------|
| Al      | 20000 ± 200                       | 12000 - 23000                   |
| As      | 90 ± 15                           | 88 - 110                        |
| Cr      | 21 ± 3                            | 15 - 25                         |
| Cu      | 90 ± 1                            | 91 - 110                        |
| Fe      | 23000 ± 2000                      | 17000 - 26000                   |
| Mn      | 600 ± 10                          | 400 - 620                       |
| Ni      | 17 ± 3                            | 14 - 20                         |
| Pb      | 1400 ± 30                         | 930 - 1500                      |
| Zn      | 300 ± 10                          | 290 - 340                       |





# Improving Detection Limits for As, Se, Hg with MSIS



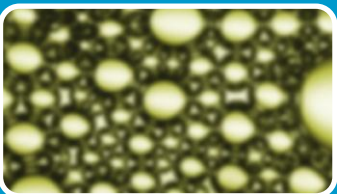
| Analyte | MSIS in Simultaneous Mode ug/L |
|---------|--------------------------------|
| As      | 1.0                            |
| Se      | 2.0                            |
| Hg      | 0.5                            |

# Application Notes Available



## Geochemical

- New methodology for analysis of gold and precious metals
- Analysis of base metals in geochemical samples



## Chemical & Petrochemical

- Analysis of wear metals and contaminants in engine oils
- Analysis of lubricating oil additives
- Analysis of trace elements in petroleum and diesel fuels



## Food & Agriculture

- Analysis of soil extracts
- Cost-effective analysis of major, minor, and trace elements in foodstuffs



## Environmental

- Determination of metals in industrial wastewaters



# Agilent 4100 MP-AES



## ***Runs on air – the most significant advance in atomic spectroscopy***

- **Lowest running cost** of any atomic spectroscopy technique due to capability to run on air – ideal for remote and at site operation
- **Improved safety** – capability to run on air means no flammable gases and no manual handling of cylinders
- **Easy-to-use** software with MP applets and plug and play torch which simplify operation and maximize uptime
- **Superior performance to flame AA**, with capability to run unattended overnight



# SVS-2 Switching Valve Accessory



**Reduced sample introduction component wear: only the volume of sample needed for analysis is delivered.**

**Faster Analysis**

**Decreased rinse times**

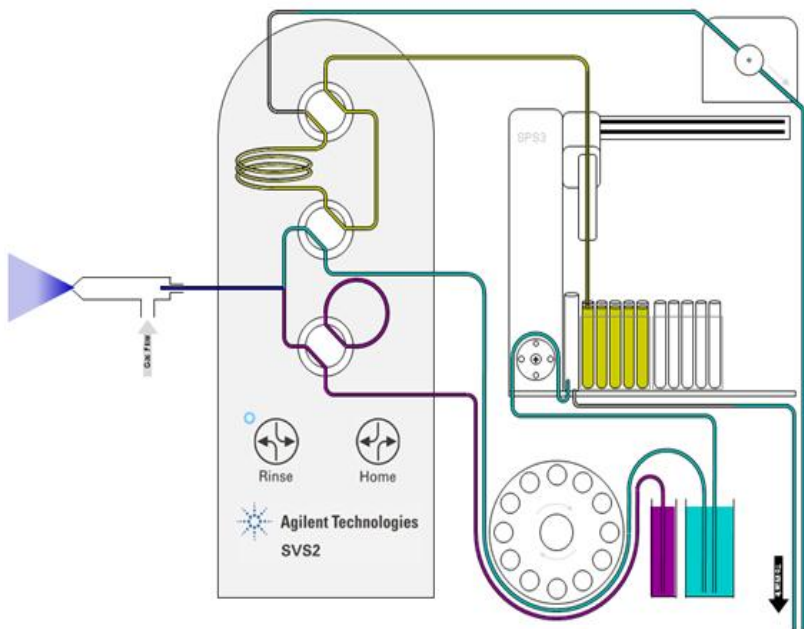
**Reduced run time = reduced run costs (argon).**

**Potential for reduced contamination as sample does not contact peristaltic pump tubing**

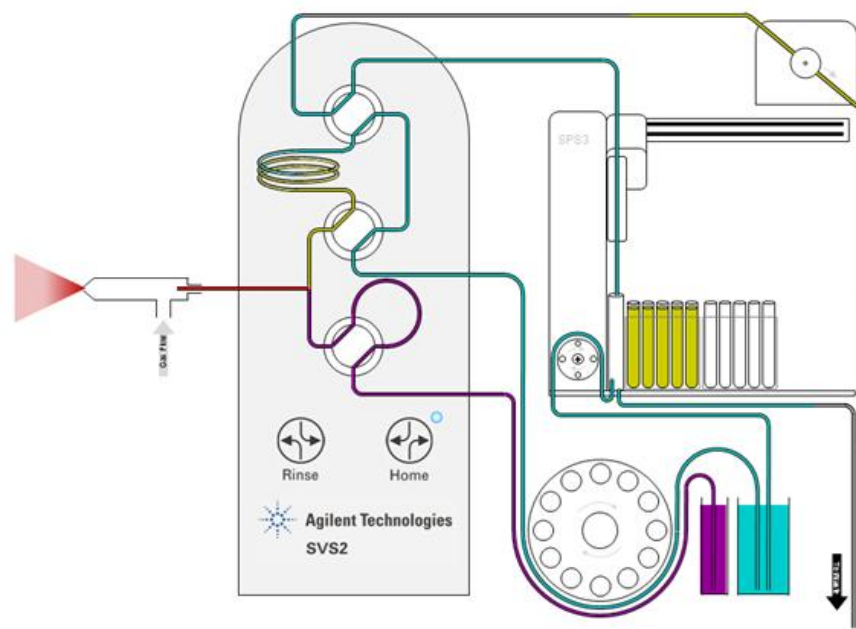
# SVS2 Hardware



# SVS-2 Switching Valve Operation

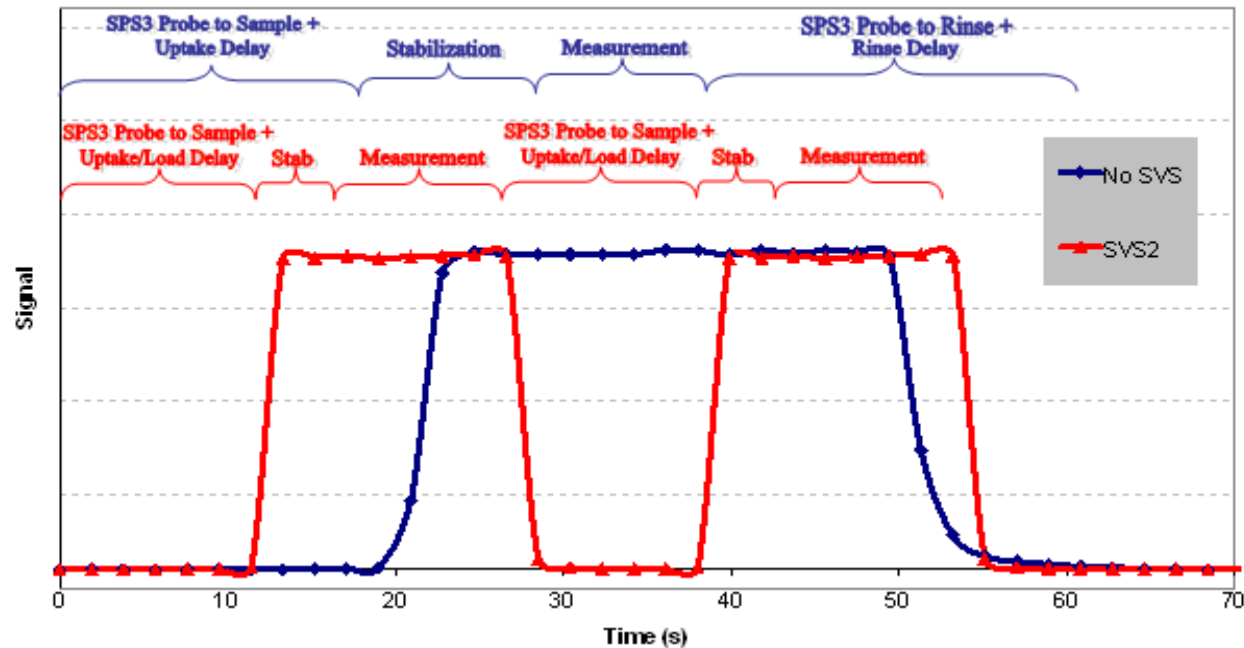


**Sample Load Position**

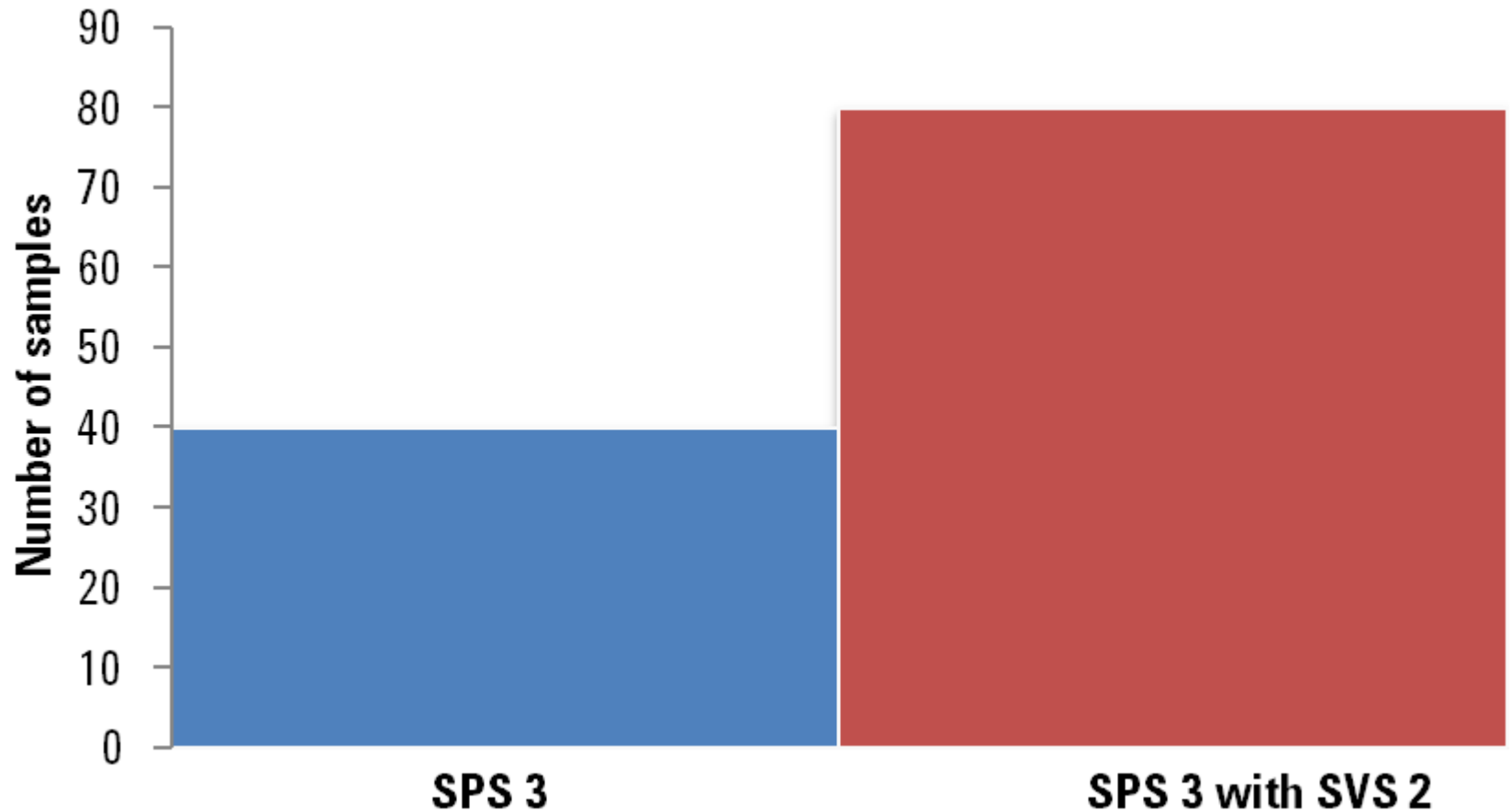


**Sample Inject Position**

# SVS-2 Switching Valve Accessory

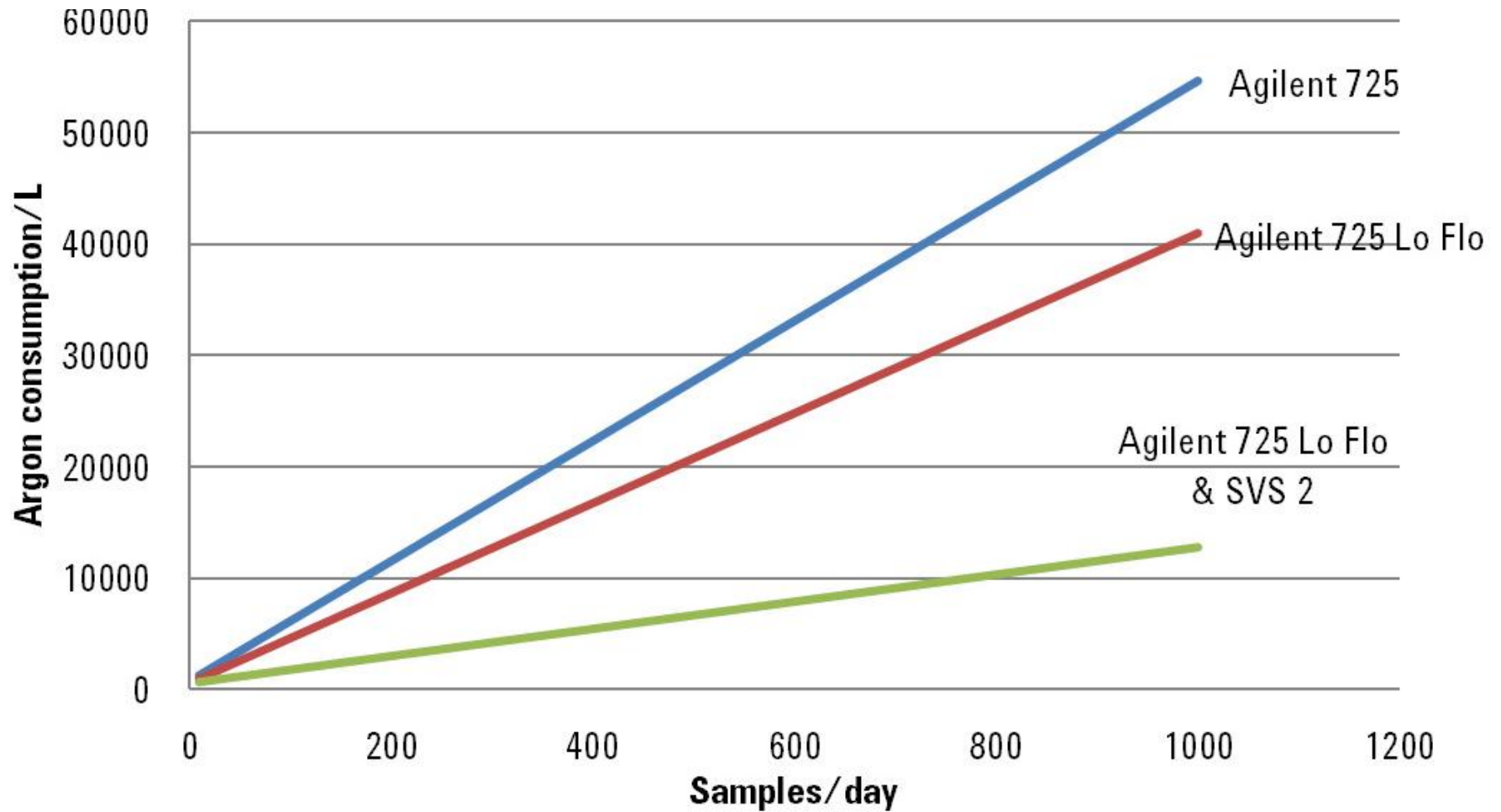


# Number of samples per hour





# Daily argon consumption



# Questions?



**Agilent MP-AES**



**Agilent AA**



**Agilent ICP-OES**



**Agilent ICP-MS**

## The Market Leaders in Atomic Spectroscopy

