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

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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_2 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

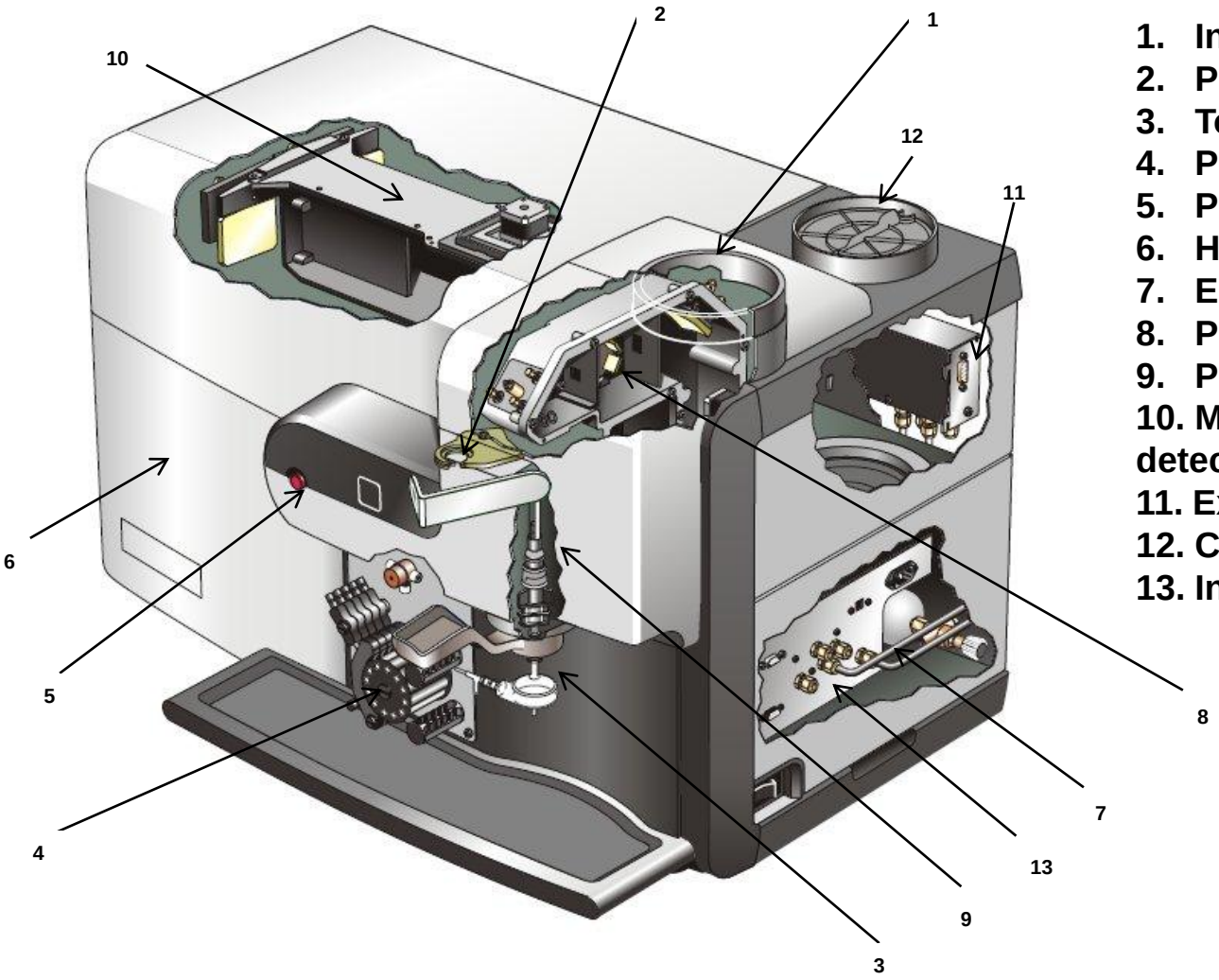
Reduced operating costs:

- 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:

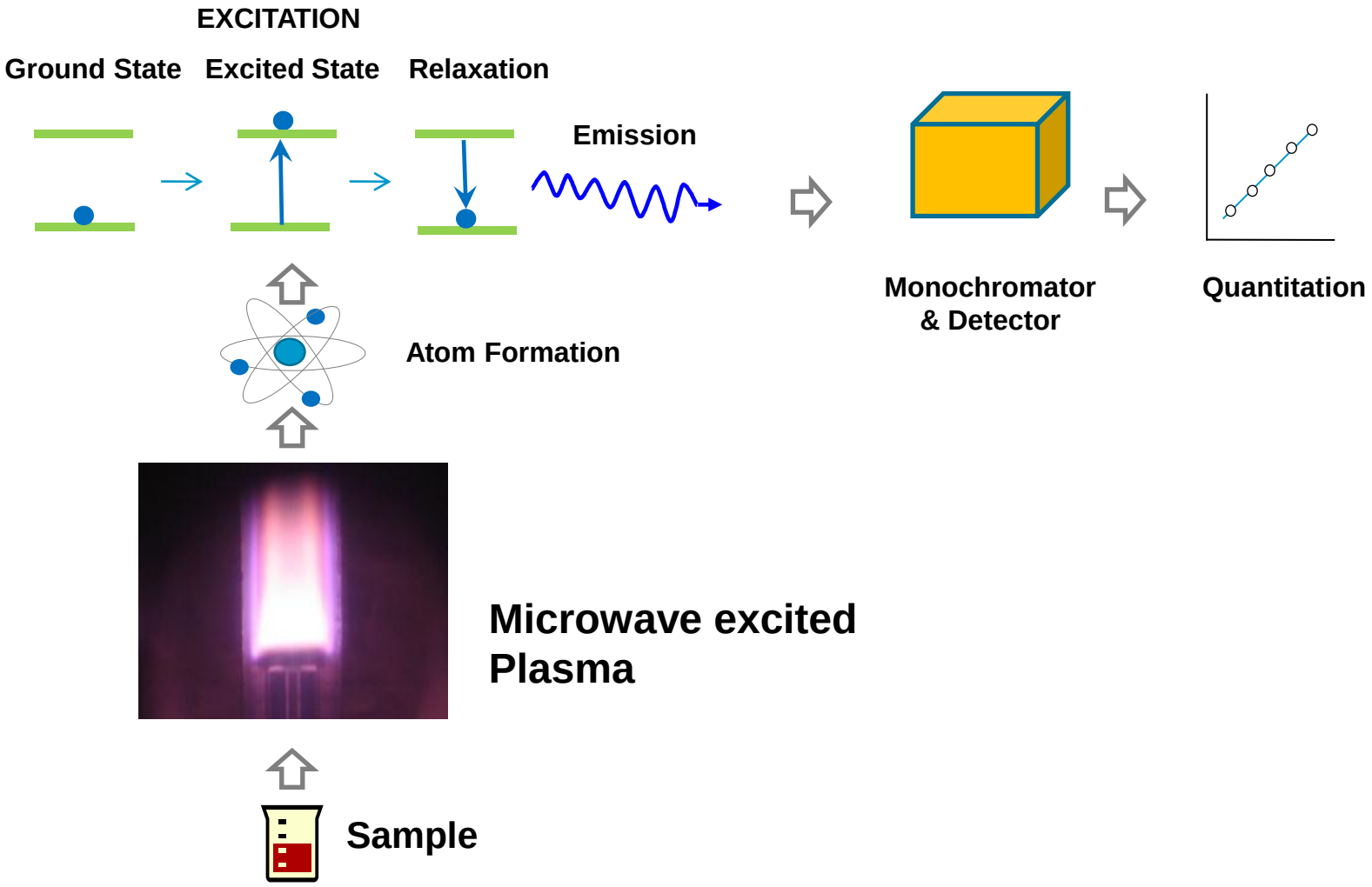
- Eliminates need for flammable gases and cylinder handling
- Safe, reliable unattended multi-element overnight operation

Schematic Diagram



1. Instrument exhaust
2. Pre-optics window
3. Torch loader
4. Peristaltic pump
5. Plasma enable button
6. High voltage power supply
7. Electronics (control PWB)
8. Pre-optics
9. Plasma *(Magnetron)*
10. Monochromator with CCD detector
11. External gas control module
12. Cooling air inlet
13. Inlet gas connections

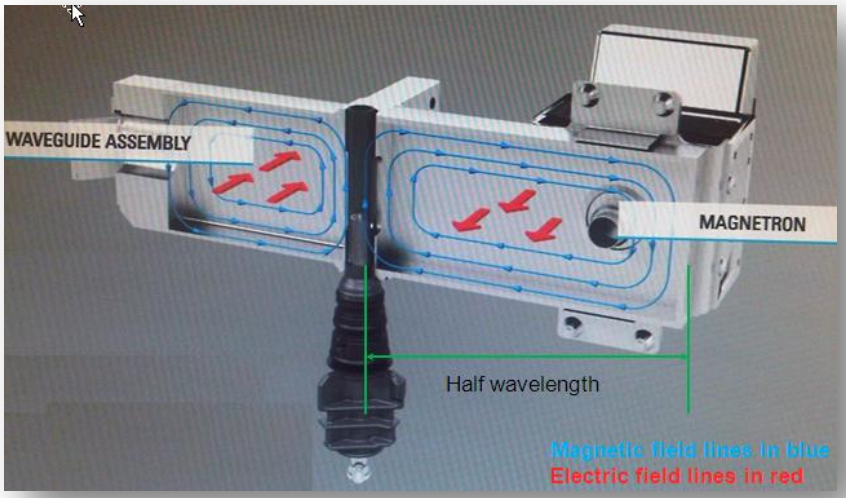
Microwave Plasma Emission Overview

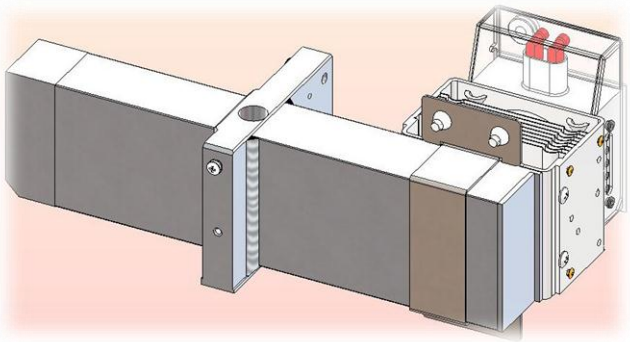


How Does MP-AES Work?

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

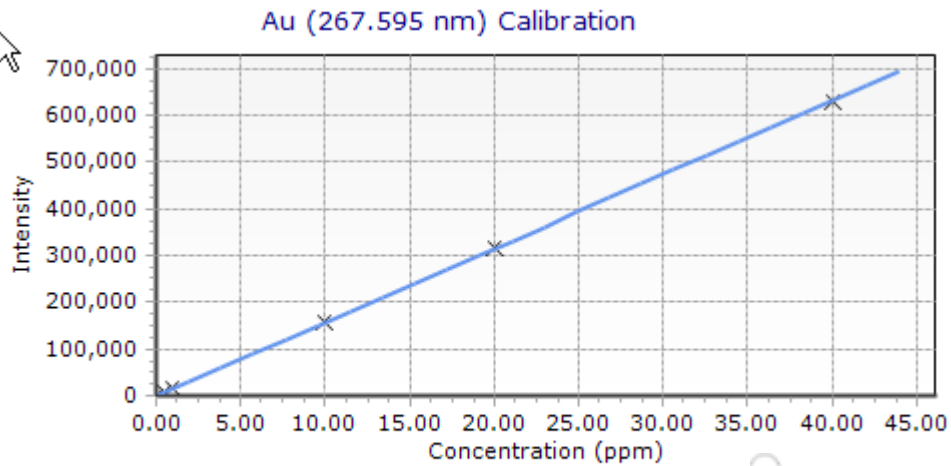
Element	Flame AA	MP-AES
K	0.8	0.65
Ca	0.4	0.05
Mg	0.3	0.12
Na	0.3	0.12
Au	5	1.8
Pt	76	4.5
Pd	15	3.8
Ag	1.7	0.5
Rh	4	0.5

Element	Flame AA	MP-AES
As*	60	45
Cd	1.5	1.4
Cr	5	0.5
Mn	1.0	0.25
Pb	14	4.4
Sb	37	12
Se*	500	70
V	20	0.2

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

3 sigma DLs using a 10 second integration time

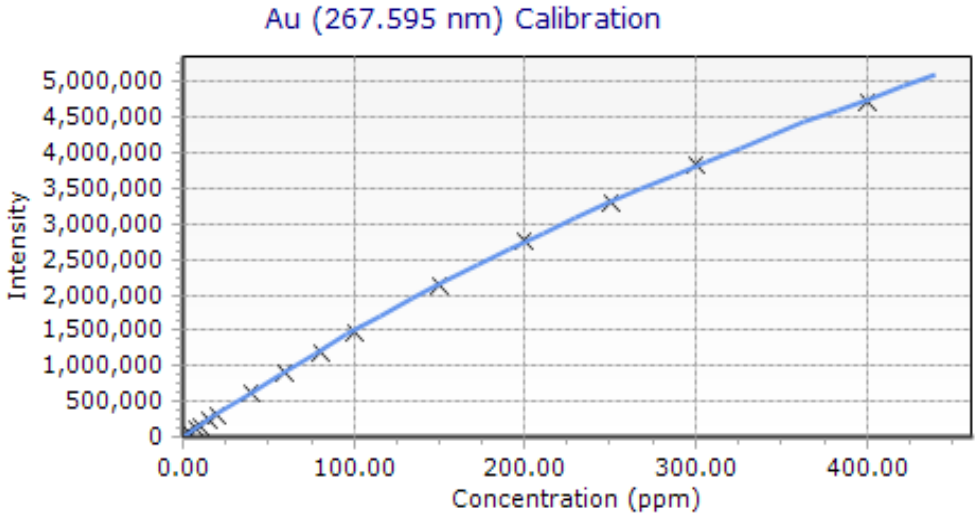
Performance – Extended Working Range



Intensity = 15775.550 * Concentration - 0.205
Correlation coefficient: 1.00000

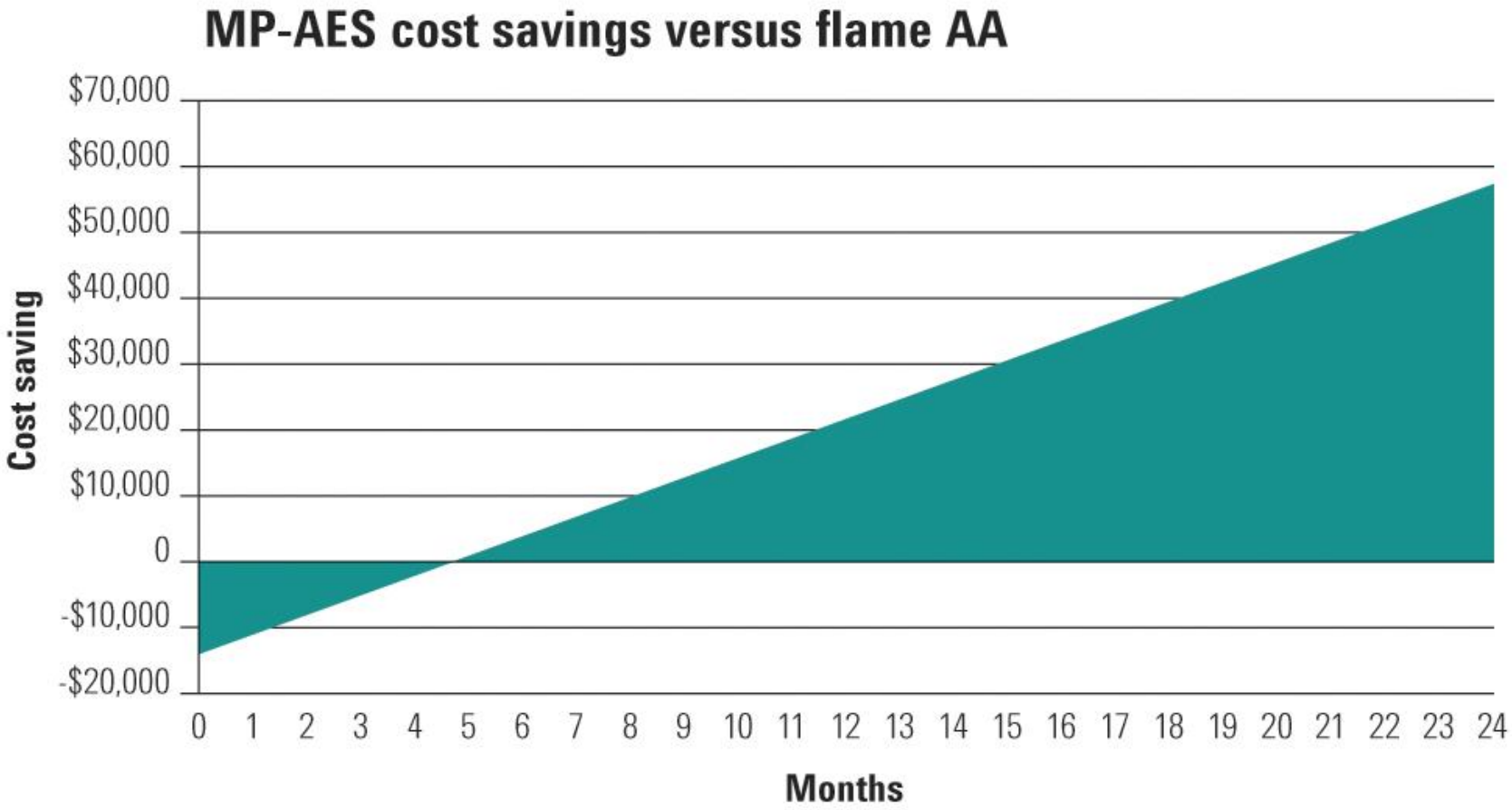
MP - normal AA range

MP - Extended Range



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

Reduced Cost of Ownership



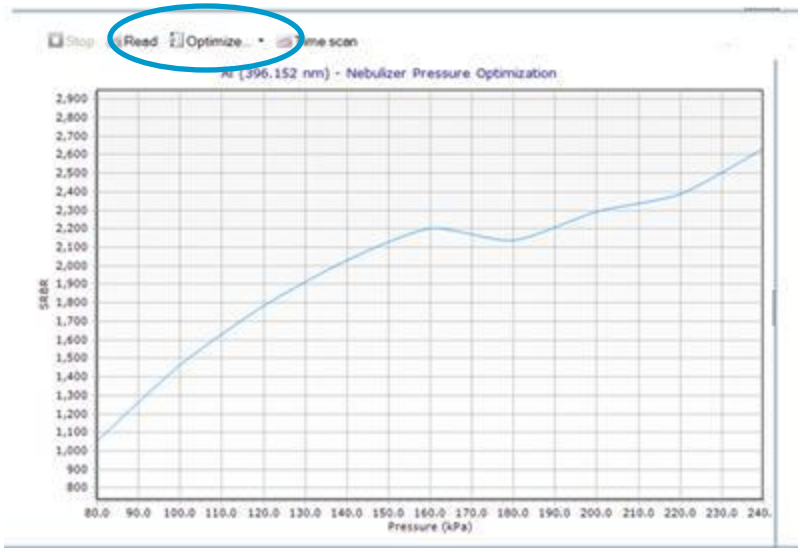
Cost savings based on reductions in gas expenses ONLY!.

Assumes measuring 9 elements (4 nitrous-oxide elements) in batches of 100 samples,
3 days/week using the N_2 generator. For a detailed cost comparison, consult your local Agilent representative

Simple Torch Installation – No Alignment

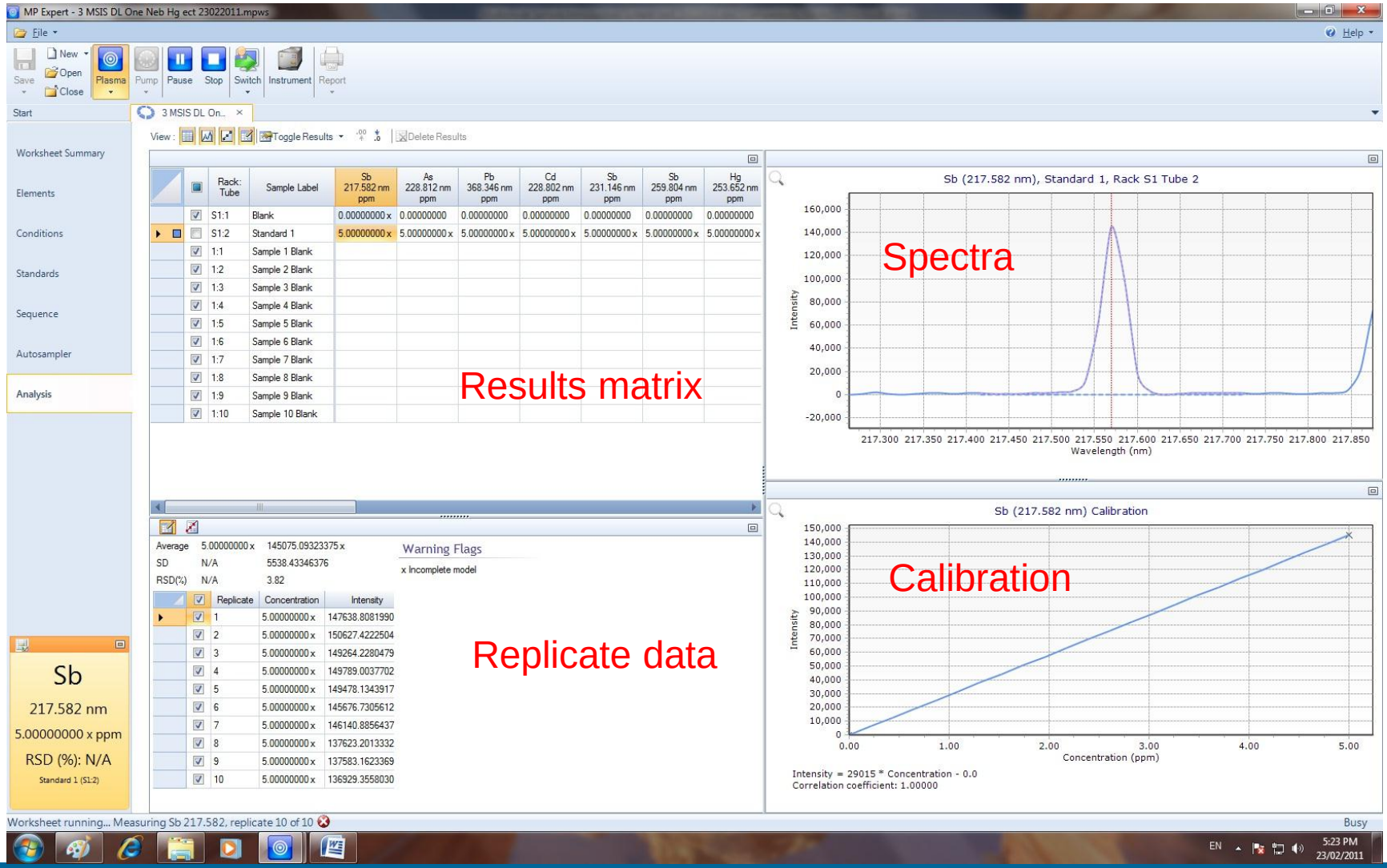
New Generation MP Expert Software

- 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



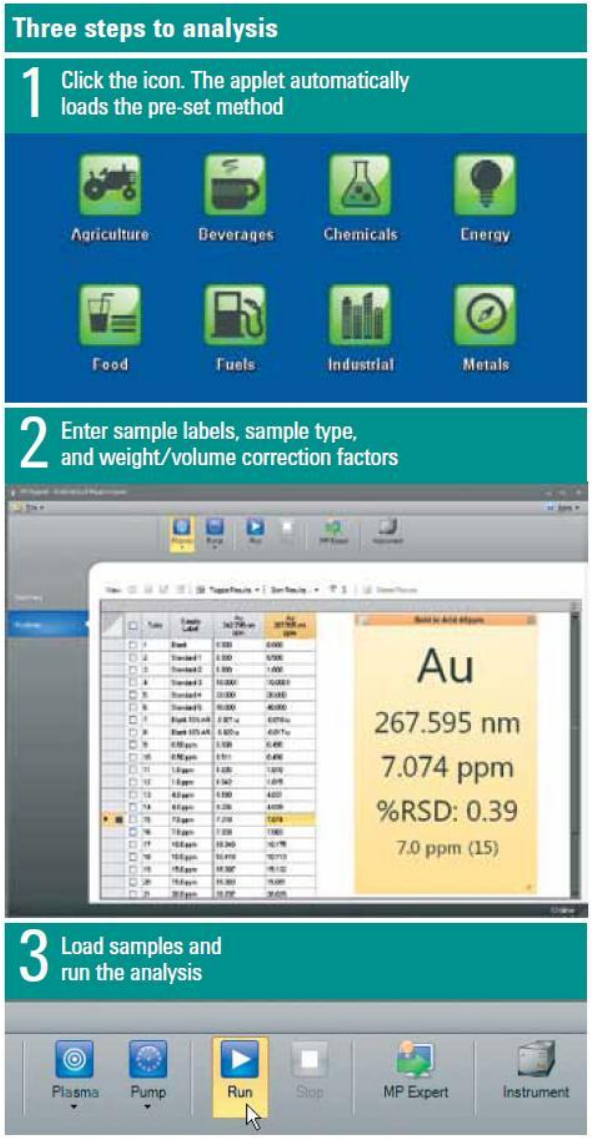
New Generation MP Expert Software

- Retains familiar “worksheet” based approach



Applet Style Operation using MP Expert Software

- Simplified software operation for operators



Accessory Options for the 4100 MP-AES

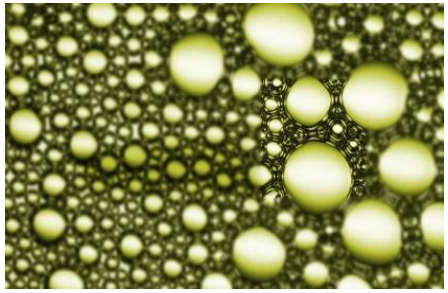
- Automate and simplify analysis with the SPS3 autosampler (required for unattended overnight operation)

- 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

Applications for the Agilent 4100 MP-AES

Geochemistry, Mining and Minerals



Chemical and Petrochemical

Food and Agriculture

Environmental

Applications MP-AES Can Do – by Market

- 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

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

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

- 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

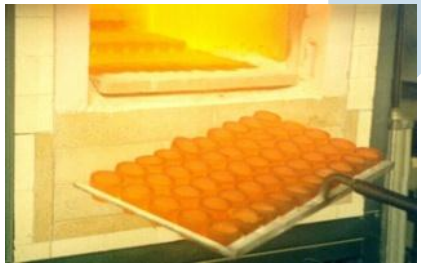
Analytical Challenges

- 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

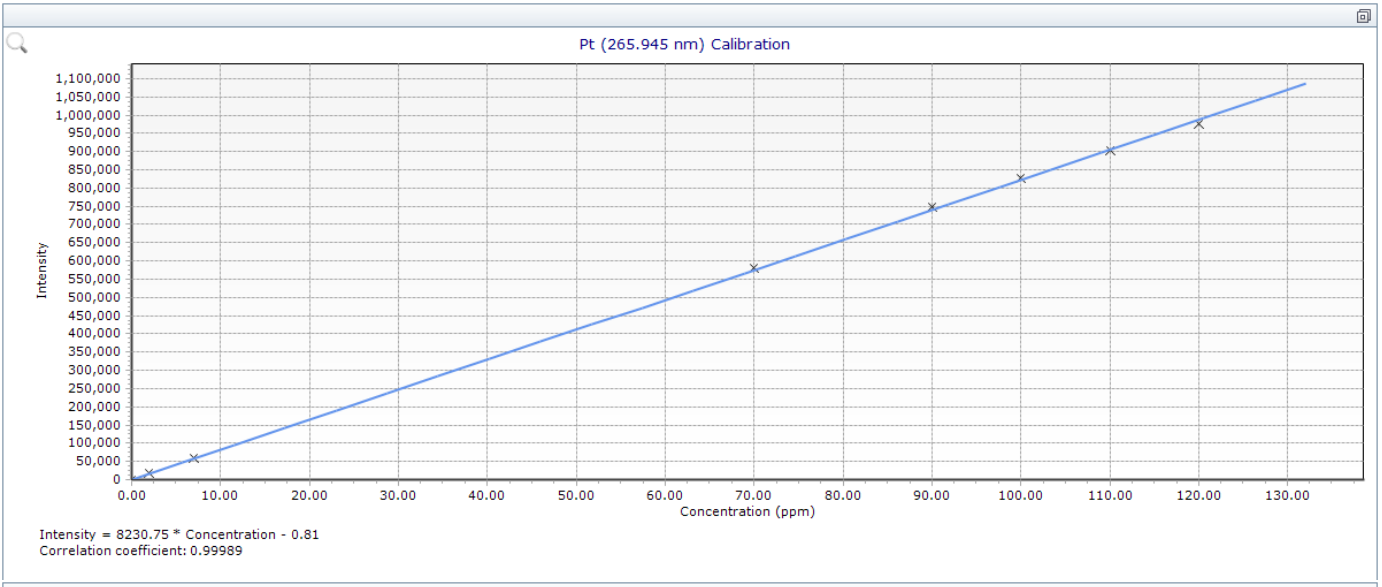
Dry and Crush → Fire Assay Process → Dissolve in 4mL 25% AR

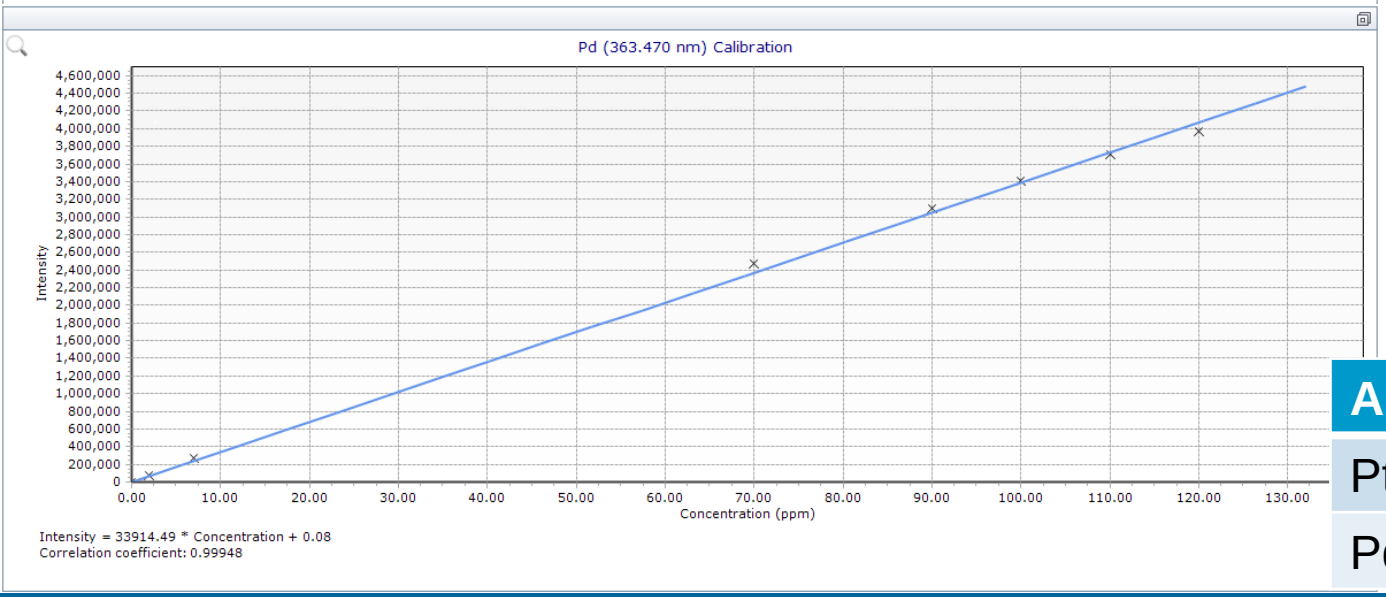


Instrument Parameters

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 = 3σ, 3s read time, 20 blanks

MP-AES Improves Throughput over Flame AA

Agilent SPS 3 Autosampler

Ore analysis can be volume limited

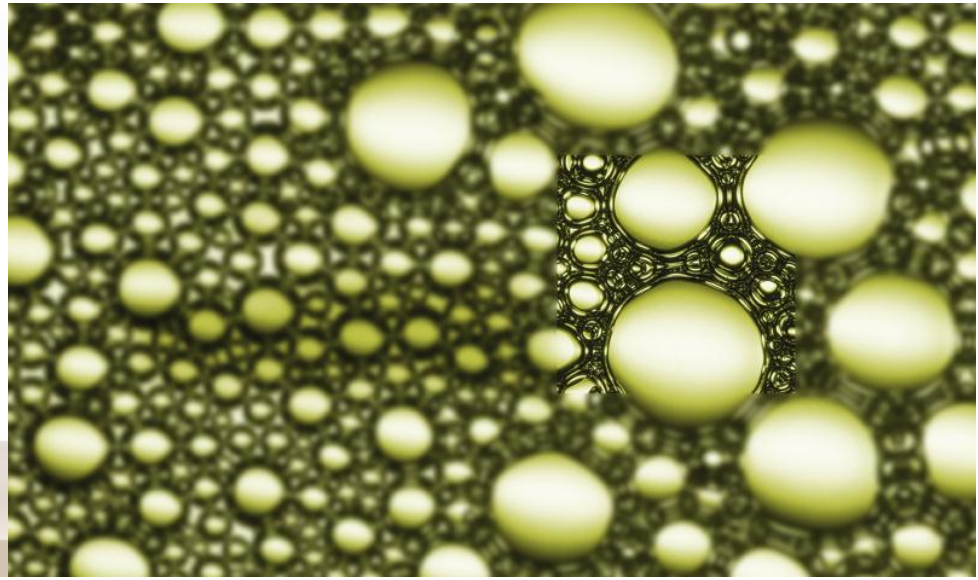
Typical sample volume available ~4mL

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

Applications for the Agilent 4100 MP-AES

Chemical and Petrochemical Applications



Applications MP-AES Can Do – by Market

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

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

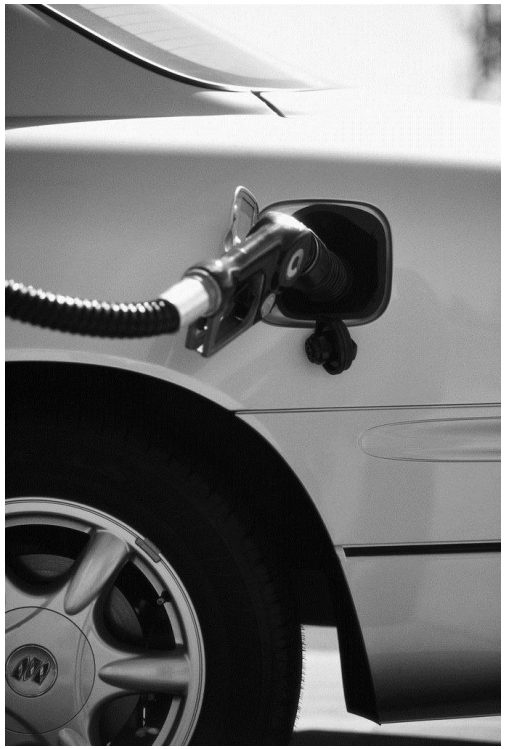
Organics Kit - External Gas Control Module (EGCM)

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

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)
Flash Point (closed cup)	**D 93**	**93 min.**	**Degrees C**
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
Water & Sediment	**D 2709**	**0.05 max.**	**% vol.**
Kinematic Viscosity, 40 C	D 445	1.9 - 6.0	$mm^2/sec.$
Sulfated Ash	D 874	0.02 max.	% mass
Sulfur **S 15 Grade** **S 500 Grade**	 **D 5453** **D 5453**	 **0.0015 max. (15)** **0.05 max. (500)**	 **% mass (ppm)** **% mass (ppm)**
Copper Strip Corrosion	D 130	No. 3 max.	
Cetane	D 613	47 min.	
Cloud Point	**D 2500**	**Report**	**Degrees C**
Carbon Residue 100% sample	D 4530*	0.05 max.	% mass
Acid Number	**D 664**	**0.50 max.**	**mg KOH/g**
Free Glycerin	**D 6584**	**0.020 max.**	**% mass**
Total Glycerin	**D 6584**	**0.240 max.**	**% mass**
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
Oxidation Stability	**EN 14112**	**3 min**	**hours**
Workmanship	**Free of undissolved water, sediment, & suspended matter**		

BOLD = BQ-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

Method Detection Limits

3σ, 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

Applications for the Agilent 4100 MP-AES

Food and Agricultural Applications

Applications the MP-AES can do – by Market

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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 % 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

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

High Purity Stds CRM Milk Powder

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

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

High Purity Stds CRM Oyster Tissue

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

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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)

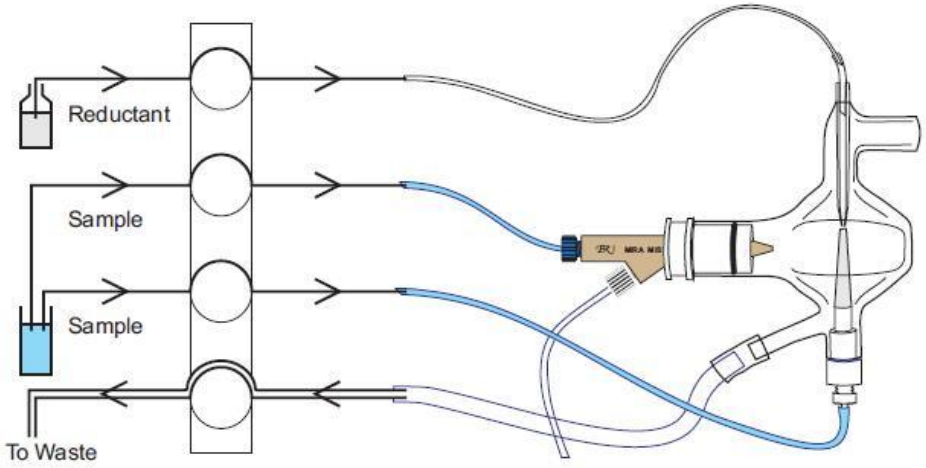
Measured Results for NIST SRM 2710 Montana Soil

Analyte	MP-AES Results Average (mg/kg)	NIST Reference 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 Average (mg/kg)	NIST Reference 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

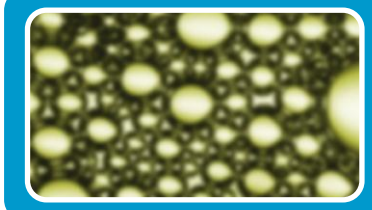


Analyte	MSIS in Simultaneous Mode ug/L
As	1.0
Se	2.0
Hg	0.5

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

Agilent's Atomic Spectroscopy Portfolio

ICP-OES

ICP-MS

ICP-QQQ

Flame AAS

Graphite Furnace AAS

4100 MP-AES

QUESTIONS?