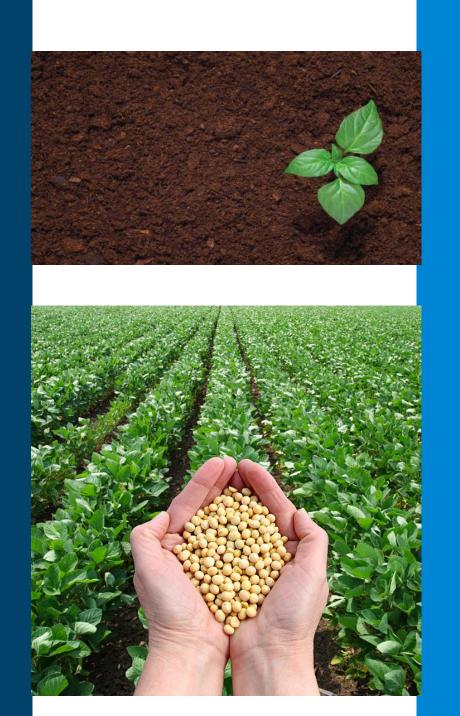
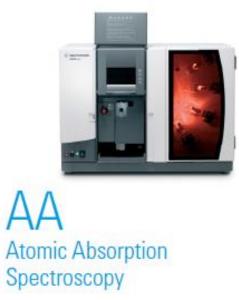
What's New in Elemental Analysis: Meet Agilent's Smart ICP-OES

ALTA Summer Meeting 2022





Agilent Atomic Spectroscopy Portfolio





MP-AES Microwave Plasma-Atomic Emission Spectroscopy



ICP-OES Inductively Coupled Plasma-Optical Emission Spectroscopy



ICP-MS Inductively Coupled Plasma-Mass-Spectrometry



ICP-QQQ Triple Quadrupole ICP-MS



ICP-OES: Ag-Related Applications

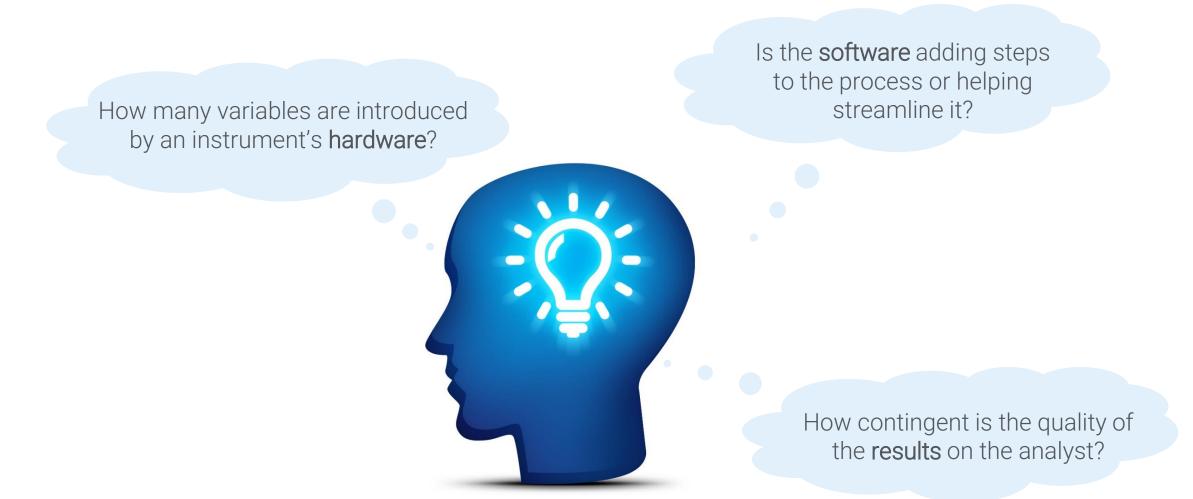
Applications Include:

- 1. Waters
- 2. Soils
- 3. Fertilizers, Fertigation
- 4. Precision Agriculture
- 5. Crops
- 6. Plants, leaves, stems
- 7. Nutrient Analysis: toxic, trace, majors
- 8. Nutritional Labeling
- 9. Packaging
- 10. Food Authenticity



Agilent Smart ICP-OES: Overview

What uncertainties exist in your lab – even before you get your results?





Hardware Smart by Design



Smart ICP-OES Hardware



Gases automatically connected

No additional manual torch optimization

No torch alignment

No User Subjectivity



Vertical Torch

- Easy-Fit
- Auto gas connections
- Auto aligned
- Design enables >25% TDS
- 5 x longer life compared to horizontal quartz torches



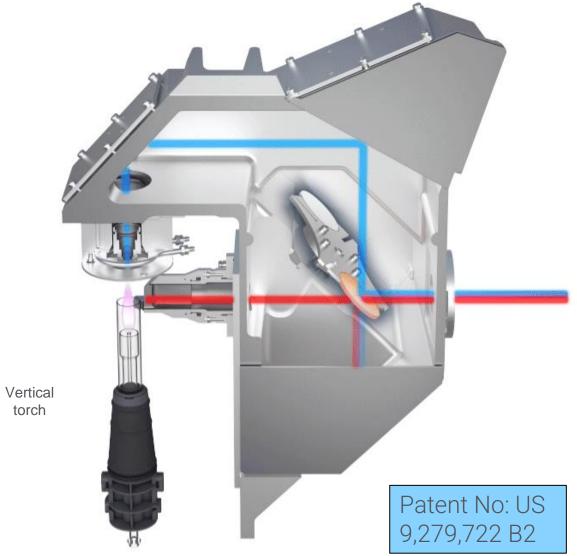
Vertical Torch

- Easy-Fit
- Auto gas connections
- Auto aligned
- Design enables >25% TDS
- 5 x longer life compared to horizontal quartz torches



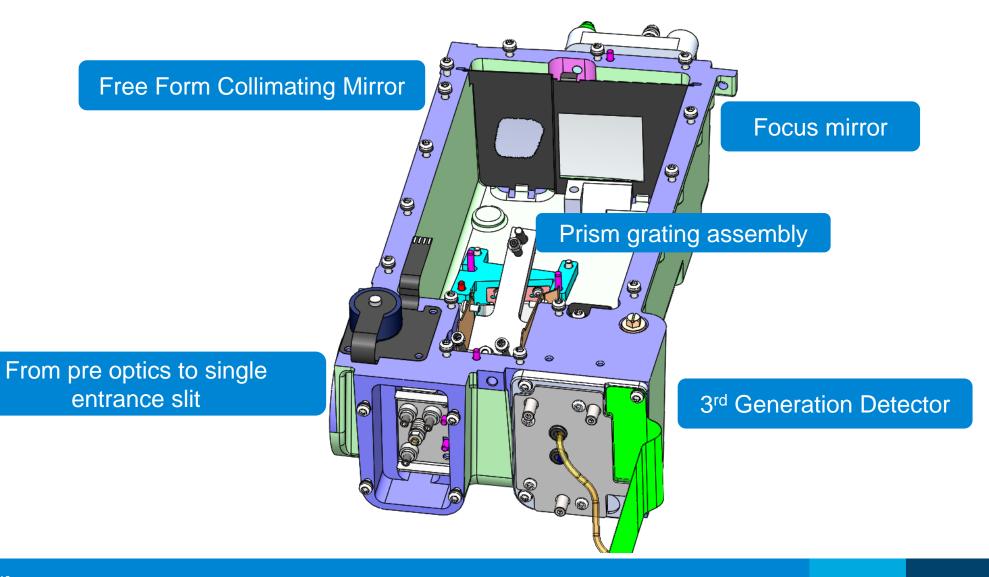


Unique Dichroic Spectral Combiner (DSC) Synchronous Technology



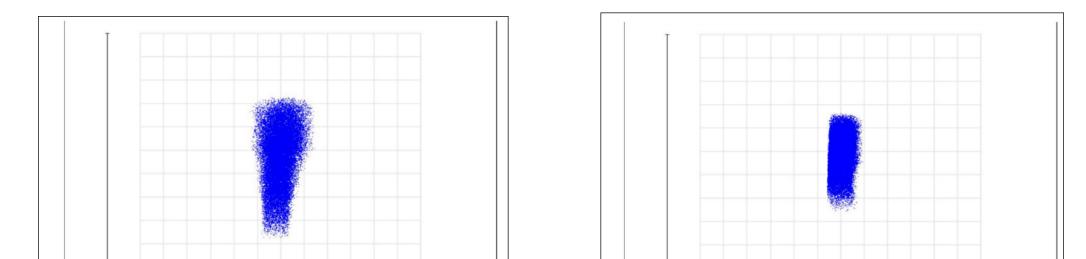


Smart Optical Design





Change to the resolution and peak shape of As188.980nm



No free form optics

Free form optics

Higher intensity

Tighter focus



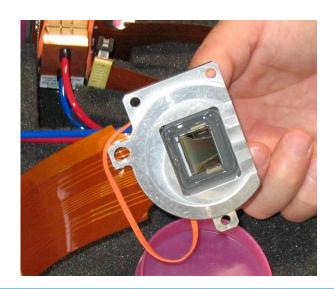
Smart Detector

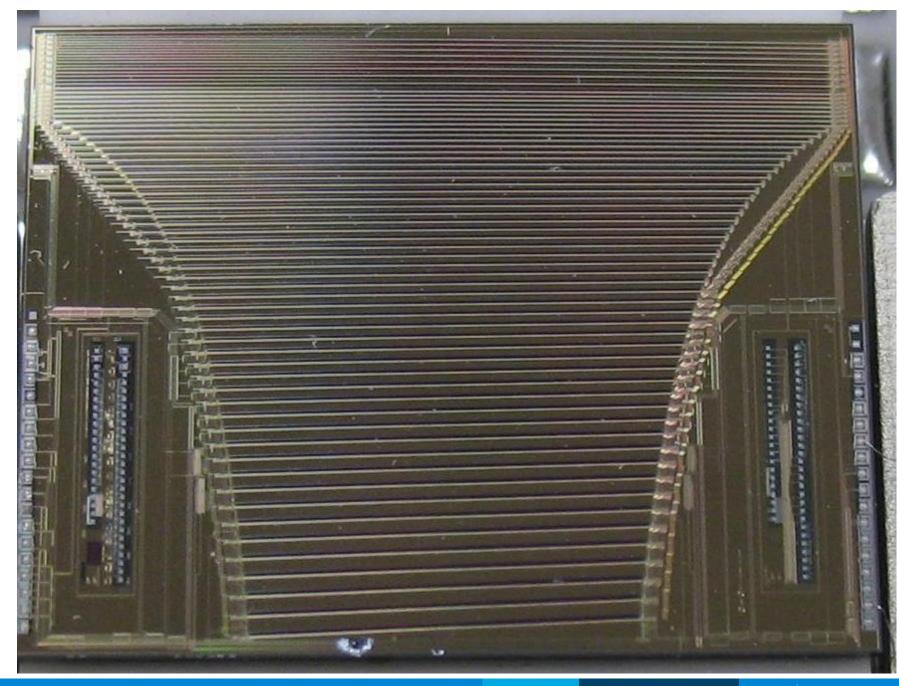
Zero gas consumption

Full wavelength coverage

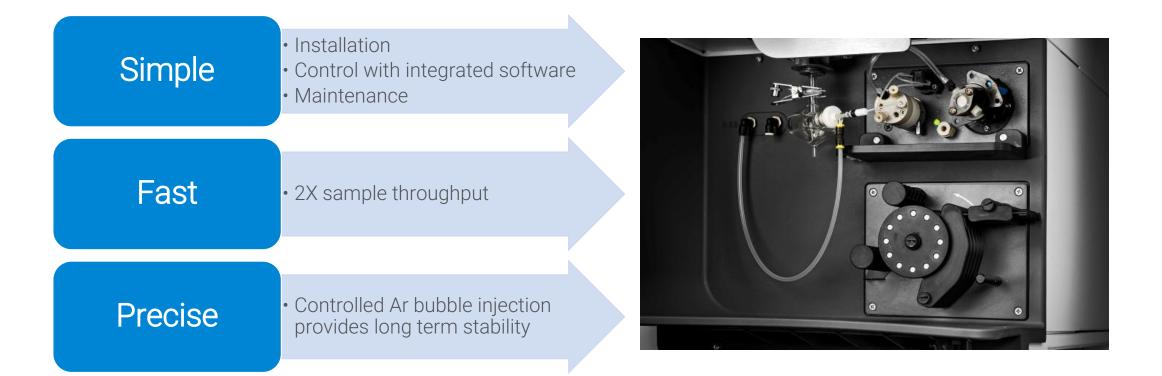
Matches image from optics

Anti-blooming





Fully Integrated Advanced Valve System (AVS) Standard in the 5900



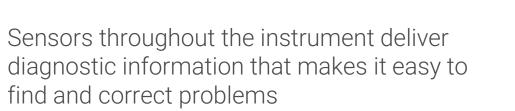
Reduce sample uptake, stabilization times, and rinse delays using the optional AVS switching valve. Controlled bubble injection for highest analytical precision.



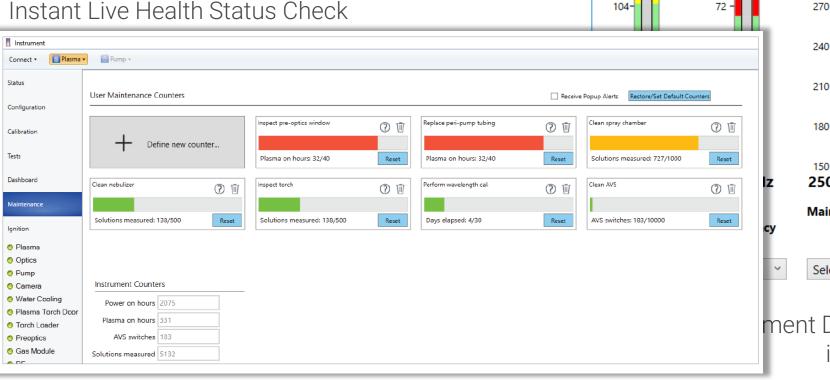
Software Smart Functionality



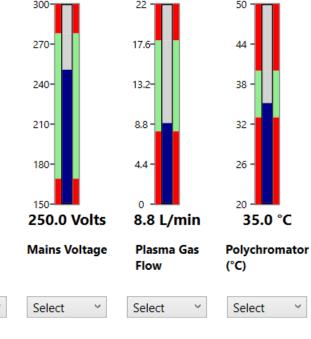
Keep Your Instrument Happy and Healthy System Diagnostics



Instant Live Health Status Check







ment Dashboard provides live information

Early Maintenance Feedback

Title

Dashboard

120-

80



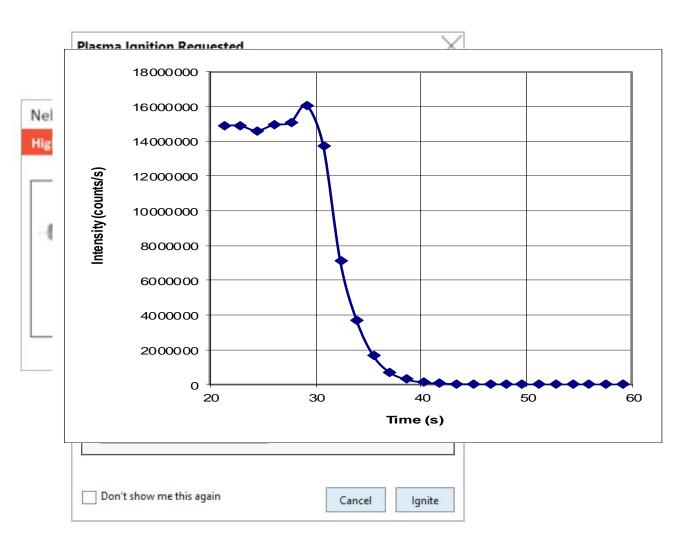
Customer Diagnostics - Tests

Status	
Confirmation	Test Result Run Tests 0
Configuration	V Poll Subsystem Test
0.11.11	Air Flow 😵 Stop
Calibration	Vater Flow
	Gas Flows Sa Export Report To PDF
Tests	RF Generator
	Camera Test
DashBoard	Optics Test
	Optical Performance
Ignition	
Detector Heating / Cooling	Use Autosampler
Plasma	Tests Run- Operator: Lindsay
Gas Module	Poll Subsystem Test-Started System Serial Number Firmware Hardware Installation Date
Diagnostics Log Config	BEAM_SELECT DEMO_BEAMSELECT 1.0 1.0 9/24/2014 2:51:00 AM RADIAL_SCAN DEMO_RADIALSCAN 1.0 1.0 9/24/2014 2:51:00 AM PUMP DEMO_PUMP 1.0 1.0 9/24/2014 2:51:00 AM SSRF DEMO_SSRF 1.0 1.0 9/24/2014 2:51:00 AM GAS_MODULE DEMO_GASMODULE 1.0 1.0 9/24/2014 2:51:00 AM
🕙 Plasma	GAS_MODULE DEMO_GASMODULE 1.0 1.0 9/24/2014 2:51:00 AM
Optics	CAMERA DEMO_CAMERA 1.0 1.0 9/24/2014 2:51:00 AM MPM DEMO_MPM 1.0 1.0 9/24/2014 2:51:00 AM Poll Subsystem Test Completed- Passed □
🙁 Pump	Air Flow-Started
Camera	Blower Frequency(%) Air Flow(Hz) Status
O Water Cooling	30% 0 - Failed 75% 0 - Failed Air Flow Completed- Failed

- Health check on instrument before analysis starts
- Can localize fault to a subsystem
- User maybe able to fix themselves
- Some automatic fixing of problems are built in

Active System Monitoring

Smart monitoring improves success for all users



Meaningful feedback with images

NebAlert nebulizer monitoring

Intelligent Rinse

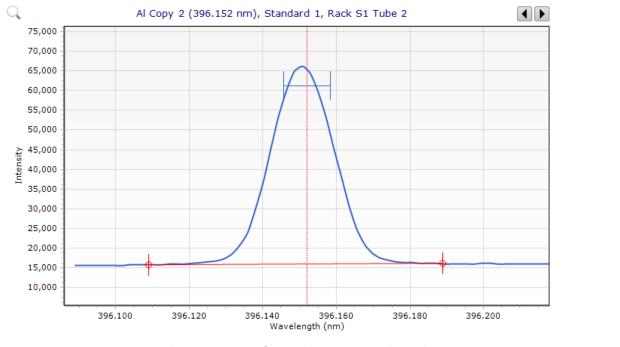


Results Sample Insights at Your Fingertips



Background Correction

FBC Fitted Background Correction: Avoid "Manual Integration"



Looks great for the standard!

Q Al Copy 2 (396.152 nm), Sample 1, Rack 1 Tube 1 360,000 340,000 320,000 300,000 280,000 260,000 240,000 220,000 200,000 ≩ 180,000 160,000 140,000 120,000 100,000 80,000 60,000 40,000 20,000 0 -20,000 396.100 396.120 396.140 396.160 396.180 396.200 Wavelength (nm)

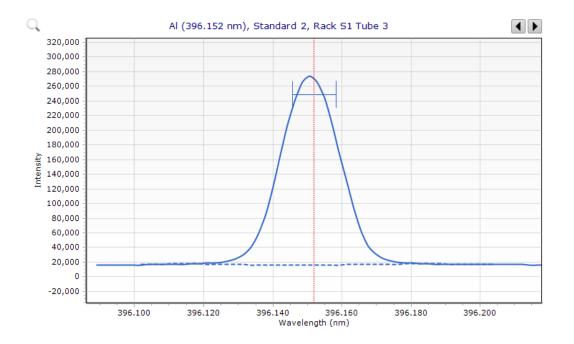
Less so for the sample....

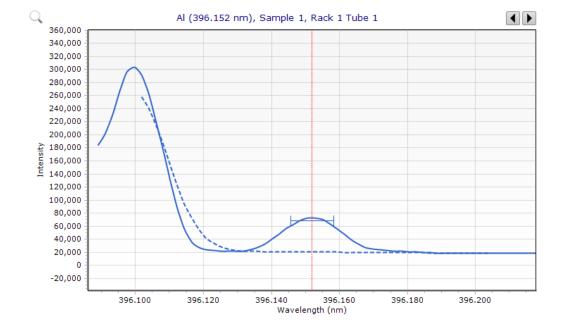
So where do the points go?



Background Correction

FBC Fitted Background Correction: Avoid "Manual Integration"





Looks great for the standard!

Better fit for the sample!

Automatically drawn by the software!



IntelliQuant

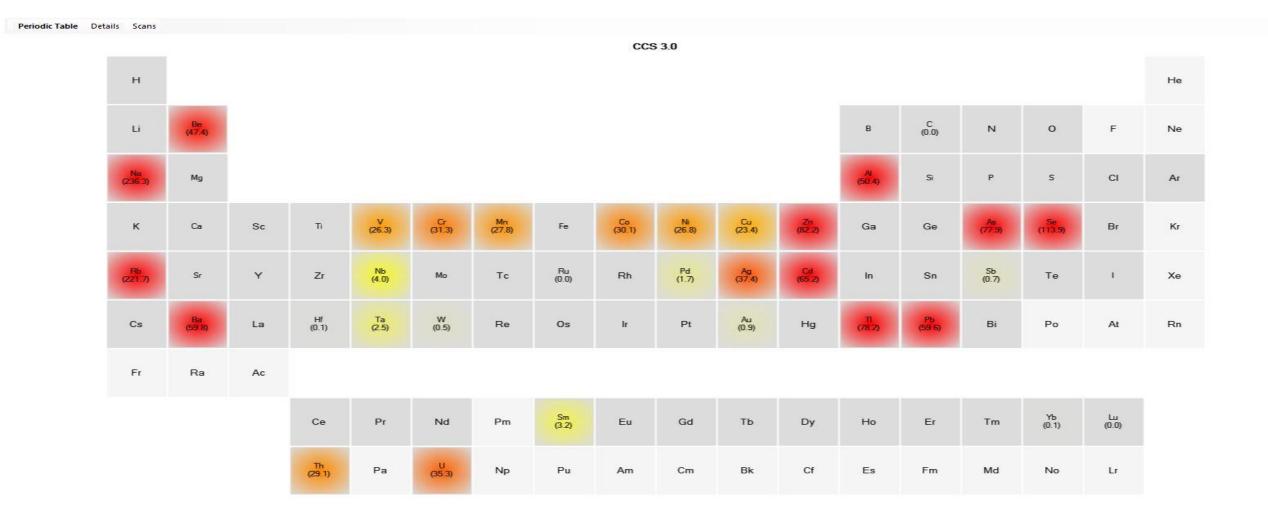
Periodic Table Det	ails Scans																	
									CCS	5 1.0								
	н																	He
	Li	Be (0.5)											B (0.5)	C (0.0)	N	0	F	Ne
	Na (2.6)	Mg (0.2)											Al (50.4)	Si (0.2)	P (75.0)	S (0.0)	СІ	Ar
	к	Ca (0.4)	Sc	Ti (0.3)	V (0.3)	Cr (0.3)	Mn (0.3)	Fe (0.4)	Co (0.3)	Ni (0.3)	Cu (0.3)	Zn (1.0)	Ga	Ge	As (0.7)	Se (1.1)	Br	Kr
	Rь (221.7)	Sr (1.0)	Y	Zr	Nb (4.0)	Mo (0.7)	Tc	Ru (0.0)	Rh	Pd (1.7)	Ag (0.4)	Cd (0.7)	In	Sn	Sb (1.1)	Te	l (0.0)	Xe
	Cs	Ba (0.7)	La	Hf (0.1)	Ta (2.5)	W (0.5)	Re	Os	lr	Pt	Au (0.9)	Hg	TI (0.8)	Рь (0.6)	Bi	Po	At	Rn
	Fr	Ra	Ac															
				Ce	Pr	Nd	Pm	Sm (3.2)	Eu	Gd	ть	Dy	Ho	Er	Tm	Үь (0.1)	Lu (0.0)	
				Th (29.1)	Pa	U (35.3)	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr	



IntelliQuant

Periodic Table Deta	ails Scans																	
									CCS	S 2.0								
	н																	He
	Li	Be											В	C (0.0)	N	0	F	Ne
	Na	Mg											AI	Si	р (75.0)	S	СІ	Ar
	к	Ca (0.3)	Sc	Ti	v	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
	Rb	Sr	Y	Zr	Nb	Мо	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	l (0.0)	Xe
	Cs	Ва	La	Hf	Ta	w	Re	Os	lr	Pt	Ац	Hg	Π	РЬ	Bi	Po	At	Rn
	Fr	Ra	Ac															
				Ce	Pr	Nd	Pm	Sm	Eu	Gd	ТЬ	Dy	Ho	Er	Tm	Υь	Lu	
				Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr	

IntelliQuant





Wavelength Selection IntelliQuant Provides Guidance

Period	dic Table		Details	Gra	ph(Pie) Graph	n(Bar) 🚽	
Element	Used	Flags	Wavelength	Rating		Concentration	Intensity	Background
Ag								
	~		328.068	****		10.63	227749.9	5799.8
			338.289	****		11.26	26201.6	1989.7
			224.641	****		7.86	1698.7	596.8
			211.383	***		8.39	990.4	622.5
AI								
			396.152	*	?	0.00	JJZ00.1	10930.6
	~		167.019	*****		8.36	46187.4	115.8
			237.312	*	?	11.03	7242.8	1414.6
			394.401	****		8.23	22691.1	6968.9
			309.271	****		7.36	24547.5	4243.9
			176.577	****		7.21	2917.9	150.2
As								
	~		188.980	****		6.83	9415.1	268.6
			193.696	****		7.31	7823.7	325.1
			234.984	***		6.99	5299.8	1295.8
			200.334	****		7.14	3093.2	428.4
			180.554	****		6.84	567.9	175.6
в								
			249.772	*	?	7.97	153342.0	1638.6
			249.678	*****	1	7.33	70424.3	1504.4

Analyte: AI(396.152)

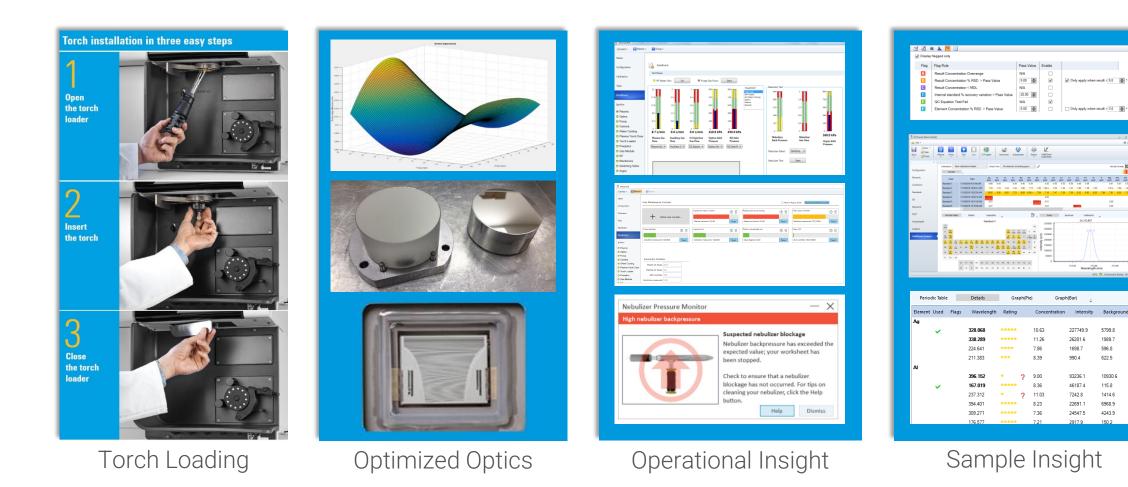
Confidence: very weak

Interference: Mo(396.151)

Confidence: very strong



A Smart ICP for a Smart Lab





Determination of Nutrients and Micronutrients in Milk Powder and Infant Formula by ICP-OES

Quality assured results by ISO 15151 using the Agilent 5800 ICP-OES with smart tools





Authors Ryley Burgess

In orderation Mice is an important source of the fats, vitamins, minerails, nutrients, and Mice is an important source of the fats, vitamins, minerails, nutrients, aurwind, mice is a source of the source of the mice is a source of the mice is a source of the source of the mice is a source is a source of the mice is a source of the mice of the mice is a source of the mice is a source fat is a source of the mice of the over detect in mice and high a source fat is a source of the and regulatory bodies towards faster, multi-element analytical techniques, such si CP-OES.





Determination of nutrients and micronutrients in milk powder and infant formula by ICP-OES

- Infant formula products –controlled by strict nutrient requirements specified by regional, national, or international, regulatory bodies.
- Inductively Coupled Optical Emission Spectroscopy (ICP-OES) provides low detection limits, high sample throughput and analytical flexibility.







Testing methods

- ISO 15151:2018 good framework for developing an ICP-OES method for manufactured milk
- Agilent 5800 ICP-OES & SPS4 autosampler used to determine Ca, Cu, Fe, K, Mg, Mn, Na, P, and Zn, in milk powder and infant formula samples according to the ISO 15151 method.





Reducing re-measurement risk

Ensuring accurate results is critical

Many food quality assurance/quality control (QA/QC) labs process large numbers of samples.

The Agilent ICP-OES Expert software includes smart tools to assist with method development, validation, and data accuracy

Smarts tools include:

- IntelliQuant
- IntelliQuant screening
- Early maintenance feedback (EMF)



Reducing re-measurement risk IntelliQuant

- IntelliQuant provides an additional semiquantitative data set for each sample
- By comparing the data sets, IntelliQuant can be used to troubleshoot the cause of any outlying data
- IntelliQuant can also be used for quality assurance of the quantitative data.

								IC	V								
н																	He
U	Be (0.6)											В	с	N	o	F	Ne
Na (102.8)	Mg (112.6)											AI (258.8)	Si (61.4)	P (60.5)	s	CI (14.4)	Ar
K (131.6)	Ca (117.6)	Sc	Ti (2.5)	V (12.0)	Cr (11.5)	Mn (5.9)	Fe	Co (24.0)	Ni	Cu (12.9)	Zn (2.4)	Ga	Ge	As (6.0)	Se (2.3)	Br	Kr
Rb	Sr	Y	Zr	Nb	Мо	Tc	Ru	Rh	Pd	Ag	Cd (2.3)	In	Sn (10.9)	Sb (23.4)	Te	L	Xe
Cs	Ва	La	Hf	Та	w	Re	Os	lr	Pt	Au	Hg	ті	Pb	Bi	Ро	At	Rn
Fr	Ra	Ac															
			Ce	Pr	Nd	Pm	Sm	Eu	Gd	Тb	Dy	Но	Er	Tm	Yb	Lu	
			Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	ſ	

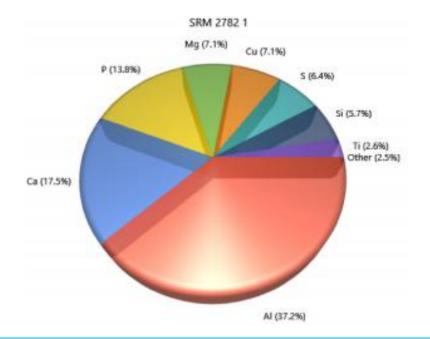
IntelliQuant allows users to visualize the content of their samples and applies color-coding to detected elements based on user-defined concentration thresholds.



Reducing re-measurement risk

IntelliQuant Screening for method development

- IntelliQuant Screening collects data without requiring selection of elements, or wavelengths, to analyze.
- IntelliQuant Screening recognizes spectral interferences—recommends the best analyte wavelengths for each element.
- IntelliQuant Screening also highlights elements in the sample that would make them unsuitable for selection as an internal standard.



A sample percentage composition chart* for user-selected elements generated in real time within the IntelliQuant Screening interface.

*Shown as an example- not representative of a milk sample.



Reducing re-measurement risk

Early maintenance feedback (EMF)

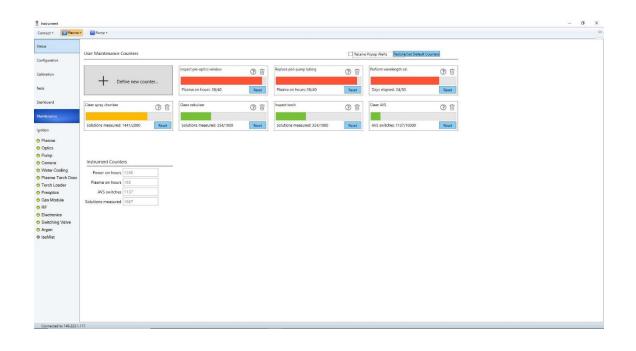
Ongoing instrument monitoring ensures that data precision and data accuracy are maintained

 Insufficient maintenance leads to costly, unplanned downtime and sample remeasurement

Maintenance that is too frequent wastes time and increases expenditure on consumables.

The EMF function includes:

- Sensors and maintenance counters that alert the user when maintenance is needed.
- Alerts for problems, and guidance for the user to take necessary action.



EMF reduces downtime and repair costs by scheduling routine maintenance of components based on actual usage, rather than at set time intervals.



Experimental Instrumentation

Agilent 5800 VDV ICP-OES with:

- SeaSpray nebulizer
- Double-pass glass cyclonic spray chamber
- Semi-demountable dual view (DV) 1.8 mm i.d. injector torch
- Agilent SPS 4 autosampler- fast and automated delivery of the samples to the ICP-OES
- An internal standard was added on-line using a Y-piece connection to the nebulizer.
- Agilent ICP Expert software

Full method parameters and instrument setup can be found in Agilent Application Note: <u>5994-2122EN</u>





Supporting Agilent's Elemental Analysis Portfolio

Application Scientists



Sima Singha

AA, MP-AES, ICP-OES

Application Scientist











Yan Cheung **ICP-MS** Application Scientist



Ana Garcia-Gonzalez AA, MP-AES, ICP-OES, ICP-MS SPSD Application Scientist

Product Specialists



Abe Gutierrez ICP-MS Product Specialist



Mark Kelinske ICP-MS Product Specialist Central US



Local Account Managers and Service Engineers

Far too many to picture on one slide... ... but all devoted to your success!

Product Specialist



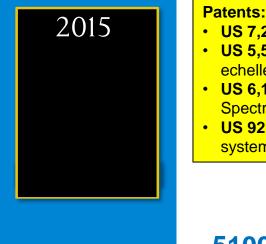
Chris Conklin Product Specialist Optical Spectroscopy





Next Options

5000 Series Awards and Innovations



nts:

- US 7,214,934 B2 RF power generator
- US 5,596,407 A Optical detector for echelle spectrometer
- US 6,137,104 A Fast Automated Spectral Fitting method
- US 92,797,722 B2 Optical emission system including dichroic beam combiner





Patents:

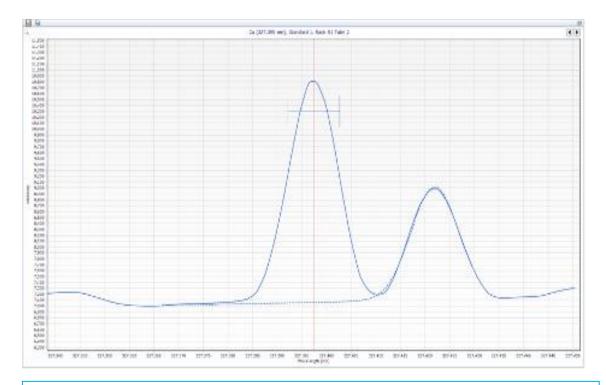
- US 94,503,30 B2 Work Coil Connection
- US 11,089,669 B2 Torch with removable injector
- WO2021/059044 A1 Variable transmission aperture
- DE112018005597T5 / CN111183342A Free Form Optics (Germany/China). Pending:
- US 2020/0379270 A1 Free Form Optics
- WO 2021/079184 A1 IntelliQuant



ICP Expert software

Background Correction

- The ICP Expert Fitted Background Correction (FBC) was used in this method.
- FBC provides background correction of both simple and complex background peaks commonly associated with the analysis of food samples.
- FBC use requires no user input or method development.



Automatic background correction using FBC for Cu: FBC provides accurate correction of the background structure arising from an OH emission line. This allows the low-level detection of Cu 327.395 nm.



Method development

- IntelliQuant Screening was used to select wavelengths and determine the approximate concentrations of each element in a sample.
- IntelliQuant Screening highlighted that strontium (Sr) was present in all the samples.
 - ISO 15151 suggests Sr as a possible internal standard for atomic lines; however, due to its presence in the samples it was not used.
- The calibration standards were prepared from Agilent single element calibration <u>standards</u> using a matrix of 20% nitric acid*.
- 18.2 MΩ de-ionized (DI) water** was used to dilute all solutions.
- A QC solution was prepared at one half of the highest concentration standard used for each element.

*Emsure, Merck Millipore **Merck Millipore



Method development

Calibration parameters

 ISO 15151 specifies the upper concentration limit for each element and that the calibration correlation coefficient for each wavelength should be >0.9995.

The 5800 VDV ICP-OES exceeded the ISO method requirements with a correlation coefficient of >0.99993 for each wavelength achieved

Element, Wavelength (nm)	Calibration Concentration Range (ppm)	ISO 15151 Upper Limit (mg/L)	Correlation Coefficient	Viewing Mode
Ca 317.933	0–350	256	0.99998	Radial
Cu 327.395	0-0.25	0.24	0.99995	Axial
Fe 238.204	0–5	4	0.99999	Axial
K 766.491	0–400	400	0.99993	Radial
Mg 280.270	0–50	22	0.99994	Radial
Mn 259.372	0-0.25	0.2	0.99999	Axial
Na 589.592	0–250	170	0.99994	Radial
P 213.618	0–250	160	0.99998	Radial
Zn 202.548	0–5	3.6	0.99991	Axial

Plasma viewing mode, background correction method, internal standard, and calibration information.

- Fitted background correction was used for all analytical lines.
- Yttrium, Y (371.029 nm) was used as an internal standard for all lines except, K, Na and P where no internal standard was used.



Results and discussion Limits of quantification (LOQ)

LOQ were based on:

- 10 σ (sigma) of 10 repeated measurements of the method blank, 20% HNO₃.
- The results of an average of six runs performed on two separate instruments.

The 5800 LOQs are all significantly lower than the lower limits specified in the ISO 15151 method

Element and Wavelength (nm)	ISO 15151 Lower Limit (mg/kg)	LOQ (mg/kg)
Ca 317.933	200	0.73
Cu 327.395	0.3	0.05
Fe 238.204	5	0.04
K 766.491	100	13.02
Mg 280.270	30	0.03
Mn 259.372	0.1	0.01
Na 589.592	100	1.83
P 213.618	150	2.39
Zn 202.548	2	0.02

The LOQ (mg/kg) for each element for the 5800 VDV ICP-OES method used.

Results and discussion CRM analysis

• Digests of three milk powder CRM samples were analyzed three times

The recoveries for all analytes with certified or reference values were within ±10% of the expected value

Element and Wavelength (nm)	Certified Value (mg/kg)	Measured Value (mg/kg)	Recovery (%)
Ca 317.933	11400	11237	99
Cu 327.395	0.52	0.51	98
Fe 238.204	2.1	2.1	102
K 766.491	17600	16886	96
Mg 280.270	1130	1089	96
Mn 259.372	0.17*	0.18	106
Na 589.592	4340	4236	98
P 213.618	9340	9740	104
Zn 202.548	42	40	96

Recoveries for certified and reference elements in CRM MP-A. n=9.

* Indicates a reference value. NA = not applicable



IntelliQuant results

Ensuring the selection of the best interference-free wavelengths leads to better-quality data.

- IntelliQuant was enabled for the quantitative analysis of the CRM digests
- Typically, semiquantitative measurements are expected to be within ±30% of the expected value.

In this study, all IntelliQuant values were within ±14% of the expected concentration

Element	IntelliQuant Measured Value (mg/kg)	Certified Value (mg/kg)	Recovery (%)
Ca	10044	11400	88
Cu	0.49	0.52	95
Fe	2.1	2.1	98
K	15899	17600	90
Mg	1083	1130	96
Mn	0.19	0.17*	114
Na	3814	4340	88
Р	9566	9340	102
Zn	44	42	105

IntelliQuant results:

Comparison of the certified and semiquantitative measured concentrations for nine of the analytes in the milk powder CRM.

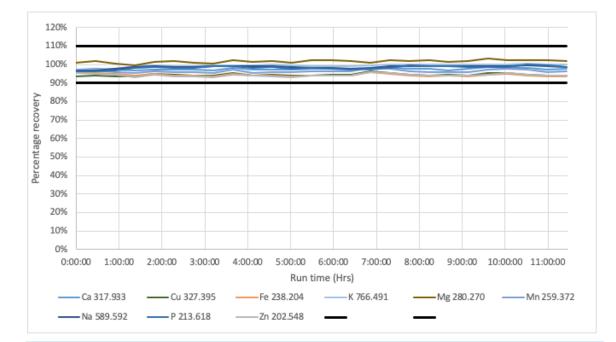
* Indicates a reference value



Long-term stability

- 250 mixed sample type solutions were measured over 11 hours without recalibration.
- A QC solution was measured after the calibration standards then after every 10 samples.

A QC stability plot shows the recovery of all elements over 11 hours to be within ±10%



Long-term stability:

Long-term stability test showing recovery of a QC solution analyzed every 10 samples over 11 hours.

Samples included digested infant formula, ready-toeat infant formula, infant formula concentrate, and milk powder samples



Long-term stability

Reproducibility of measurements

- Over 11 hours the relative standard deviation (%RSD) was less than 0.9% for all elements.
- The instrument was stable, resulting in no QC failures.
- No samples required re-measurement.

Results show excellent robustness and precision of the 5800 ICP-OES for the routine QC analysis of milk powder samples

Element and Wavelength (nm)	Concentration (ppm)	%RSD
Ca 317.933	175	0.5
Cu 327.395	0.125	0.8
Fe 238.204	2.5	0.7
K 766.491	200	0.9
Mg 280.270	25	0.8
Mn 259.372	0.125	0.8
Na 589.592	125	0.7
P 213.618	125	0.9
Zn 202.548	2.5	0.8

Long-term stability:

Concentration and %RSD of the QC sample analyzed periodically throughout the long-term stability test.



Conclusion

Agilent 5800 VDV ICP-OES with an SPS 4 autosampler can do accurate, routine measurement of elements in milk powder and infant formula, carried out in accordance with the ISO 15151 method.

This was demonstrated by:

- LOQs determined to be below the lower limit requirements for ISO 15151.
- The calibration range and linearity showed that the upper limits specified in the method could be easily achieved.
- Recoveries for the nine elements in a milk powered CRM, all being within ±10% of the certified or reference values
- The system maintaining excellent stability over the 11-hour QC recovery test



Conclusion

During the analysis smart tools were used to assist with method development and result validation.

These tools:

- Reduced the need to re-analyze any samples
- Increased confidence in the quantitative results

The use of EMF was highlighted as a tool to maintain instrument performance and avoid downtime during the analysis





Questions?





References

- 1. ISO 15151:2018 [IDF 229:2018], Milk, milk products, infant formula and adult nutritionals Determination of minerals and trace elements—Inductively coupled plasma atomic emission spectrometry (ICP-AES) method, accessed June 2020, <u>https://www.iso.org/standard/70900.html</u>
- 2. Agilent ICP Expert Software: Powerful software with smart tools for ICP-OES, Agilent publication, <u>5994-1517EN</u>
- 3. Agilent IntelliQuant Software: For greater sample insight and simplified method development, Agilent publication, <u>5994-1516EN</u>
- 4. Agilent IntelliQuant Screening: Smarter and quicker semiquantitative ICP-OES analysis, Agilent publication, <u>5994-1518EN</u>
- 5. Method for determination of silicone oil leakage and one-time sublimation and drying end point in freeze dryer, Chinese patent CN10223705, issued 2016, Accessed June 2020, <u>https://patents.google.com/patent/CN105784289A/en</u>

Note: The Agilent 5800 VDV ICP-OES requires the optional ICP Expert Pro pack software to access IntelliQuant Screening. ICP Expert Pro pack software is standard with the Agilent 5900 SVDV system and is also recommended for this application.

