

# Rapid Sample Assessment and Assisted Method Development for ICPOES Analysis Using the Agilent IntelliQuant Tool

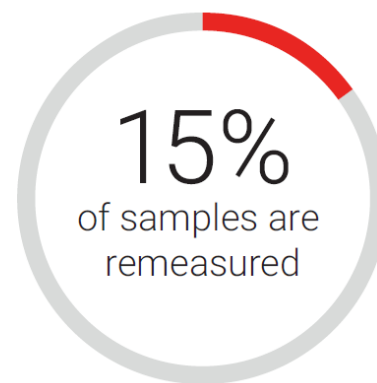
National Environmental Monitoring Conference 2020  
Metals Analysis and Remediation Session  
7 August, 2020

Greg Gilleland  
Application Scientist, Optical Spectroscopy  
Agilent Technologies



## All labs remeasure some of their ICP-OES samples. Do you know how much remeasurement is costing you?

A recent online poll showed the average amount of remeasurement is 15%.

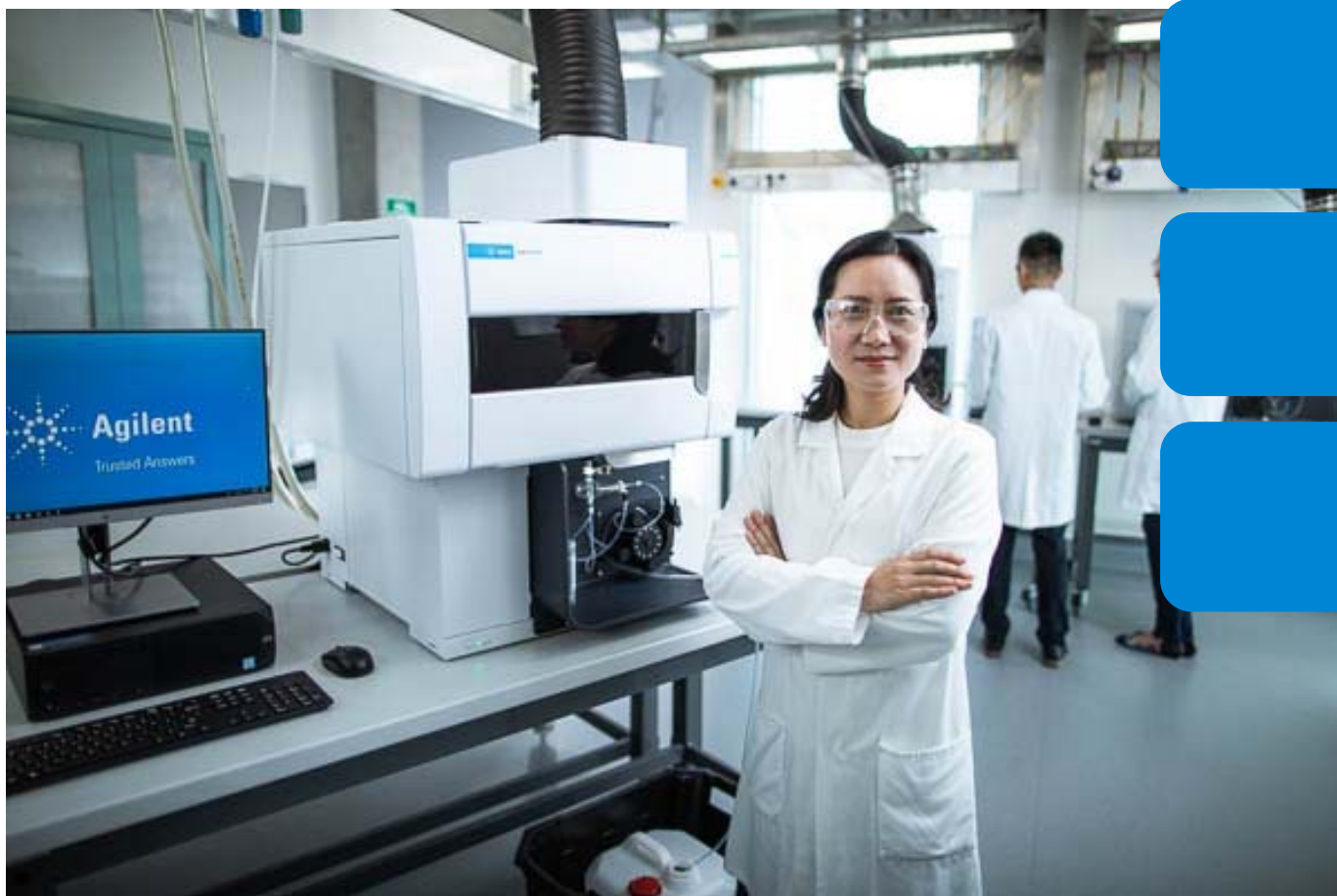


For a small to medium lab\* this will consume almost 2 additional weeks per year



\* 250 samples per week, 2.5 minutes ICP-OES analysis time per sample

## Agilent Smart Tools reduce your remeasurement



Intelligent

Efficient

Confident

# What issues lead to sample remeasurement?

Sample Related

Operation Related



## 5800 ICP-OES introduces smart tools to reclaim your wasted time

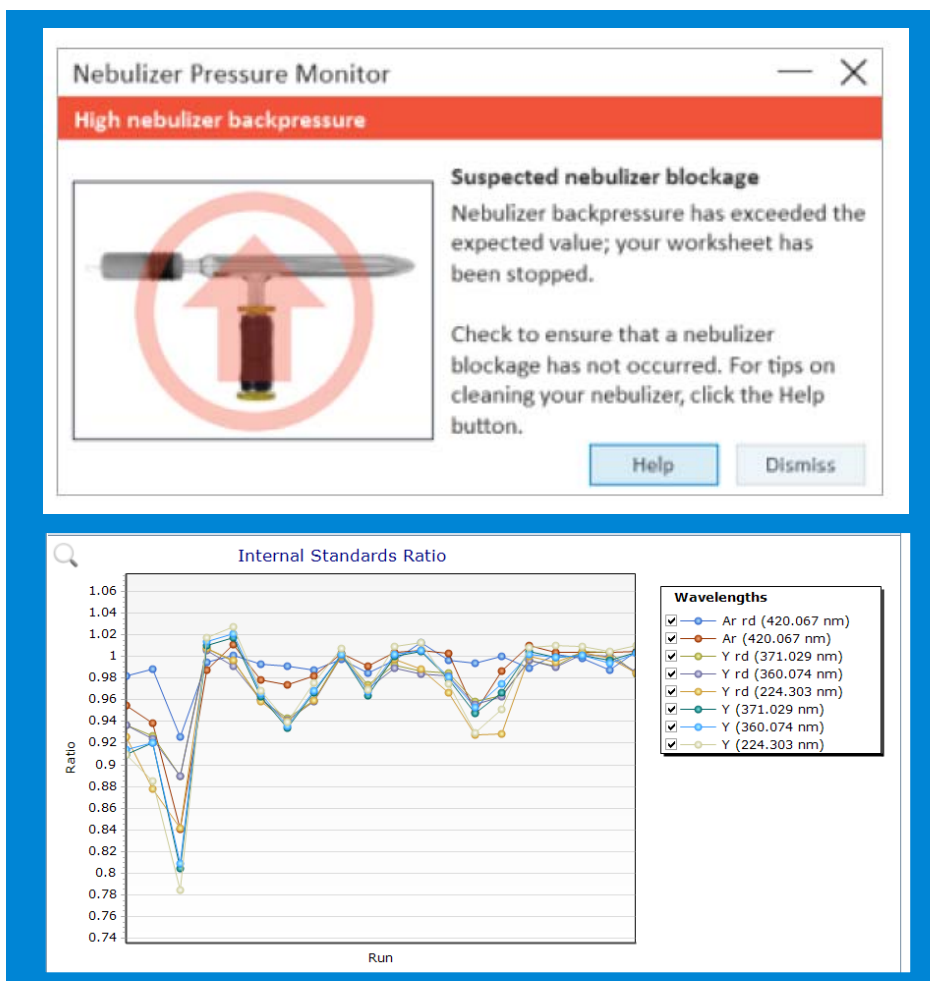
Use Smart Tools to gain insights into your **instrument operation** to reduce remeasurement

- NebAlert
- Outlier Conditional Formatting (OCF)
- Early Maintenance Feedback (EMF)
- Automated air filter blockage alert
- Automated instrument performance tests
- Intelligent rinse
- Plasma ignition optimization
- Internal standard monitor

Smart Tools

Operational Insights

# Smart tools for operational insight



NebAlert

Internal Standard monitor

# Smart tools for operational insight

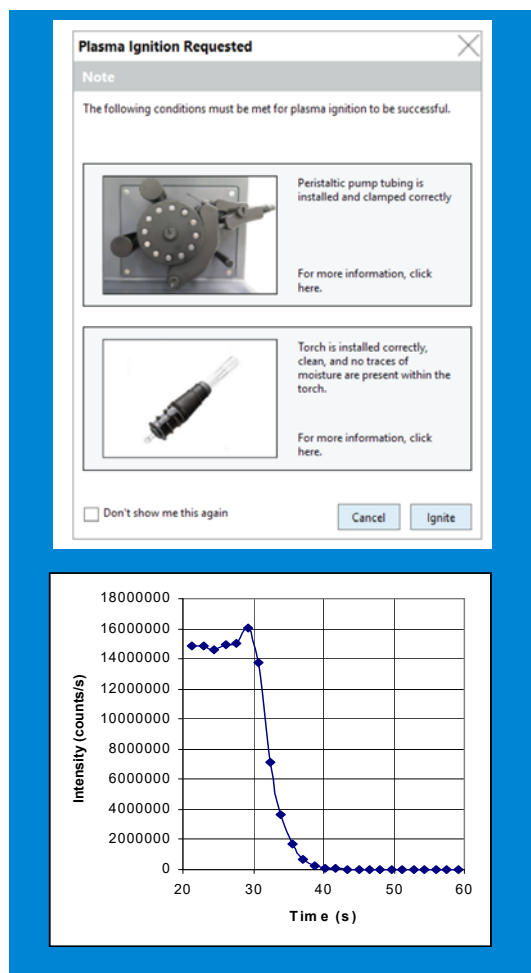
Rack	Table	Solution Label	Outlier Summary	Co	Co	Co	Co	Co	K	La	Li	M
				ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
2.2		ORESA 45x 2		1.3645	1.1663	19.6747	14.8628	3340.6496	57.8891	0.0167	0.1325	7
2.3		SRM 2781.1		0.2291	0.1247	3.7985	11.3385	520.5713	93.9258	0.3927	0.1407	107
2.4		SRM 2781.2		0.2323	0.1270	3.8096	11.8304	531.5768	97.1176	0.3894	0.1445	109
2.5		SRM 2782.1	A	1.5177	1.4996	2.3091	53.5324	5852.2018	71.4071	1.2246	0.1355	55
2.6		SRM 2782.2		1.4880	1.4333	2.1943	61.4177	6474.6380	47.6444	1.1696	0.1378	43
2.1		ORESA 45x 1		1.4382	1.2215	20.2531	15.9887	3345.5674	61.0564	0.0947	0.1407	4
2.2		ORESA 45x 2		1.4042	1.1999	20.8033	15.1864	3419.9182	59.4211	0.0168	0.1520	7
2.3		SRM 2781.1		0.2290	0.1244	3.8020	11.4742	534.8713	96.1179	0.3911	0.1410	110
2.4		SRM 2781.2		0.2405	0.1336	3.9748	12.0963	650.3115	100.0958	0.4072	0.1410	112
2.5		SRM 2782.1	A	1.5041	1.4722	2.2020	50.6637	5622.7952	71.1060	1.2091	0.1204	55
2.6		SRM 2782.2		1.4680	1.4463	2.2334	51.6804	5436.4548	66.9064	1.1841	0.1276	51
2.1		ORESA 45x 1		1.4573	1.2448	20.8513	16.0516	3447.9631	62.1334	0.0953	0.1377	4
2.2		ORESA 45x 2		1.4048	1.2017	20.1368	15.2630	3419.2966	59.4457	0.0166	0.1269	7
2.3		SRM 2781.1		0.2334	0.1267	3.8933	11.6111	531.2126	98.6493	0.3968	0.1374	113

Outlier Conditional Formatting (OCF)

Instrument Maintenance Counters (EMF) interface showing various maintenance tasks and their completion status.

Early Maintenance Feedback (EMF)

# Smart tools for operational insight



Optimized plasma ignition

Intelligent Rinse



# What issues lead to sample remeasurement?

Sample Related

Operation Related



## Smart tools that reduce your remeasurement

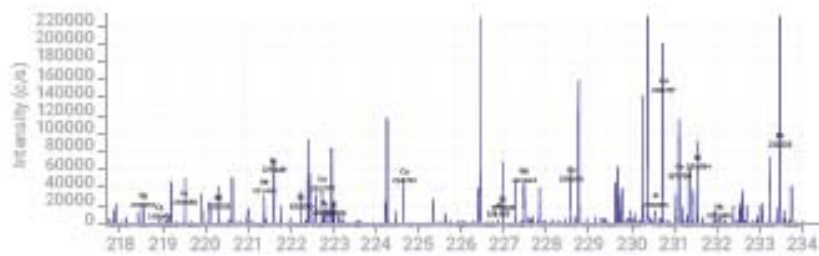
Use Smart Tools to gain **insights into your samples**

- IntelliQuant 2.0
  - Full spectrum scan
  - Identify sample preparation and chemical compatibility mistakes
  - Automated spectral interference identification
  - Understand sample composition
- IntelliQuant Screening
- Internal Standard Monitor
- Argon Emission Monitor

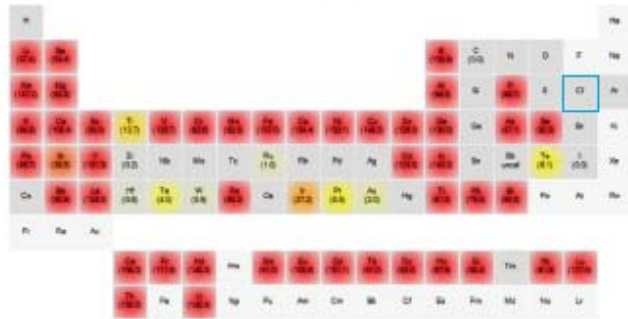
Smart Tools

Sample Insights

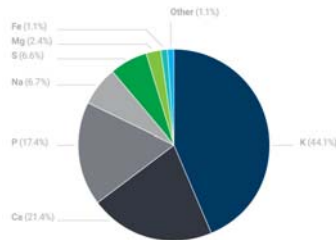
# IntelliQuant 2.0 features to help reduce re-measurement



Full spectrum scan shows everything that is in the sample



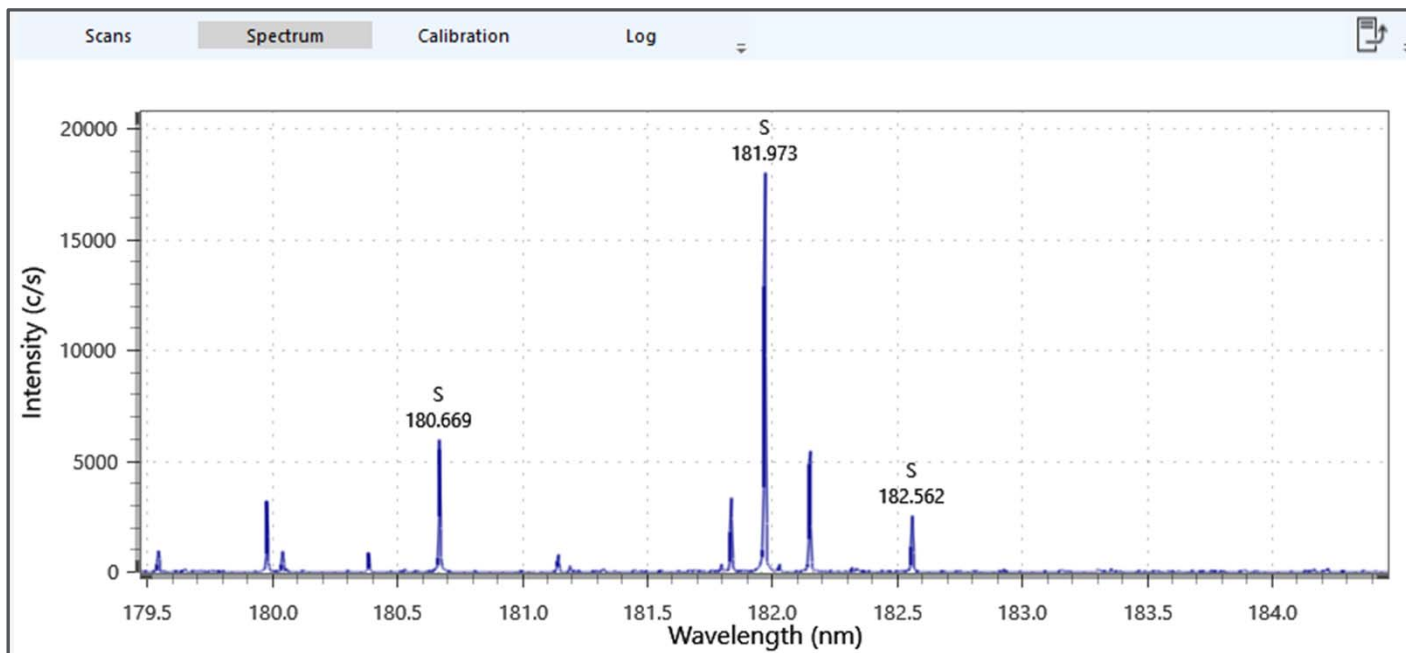
Periodic table heat map with semi-quant values to quickly identify sample preparation mistakes.



Smart view to identify composition of a sample

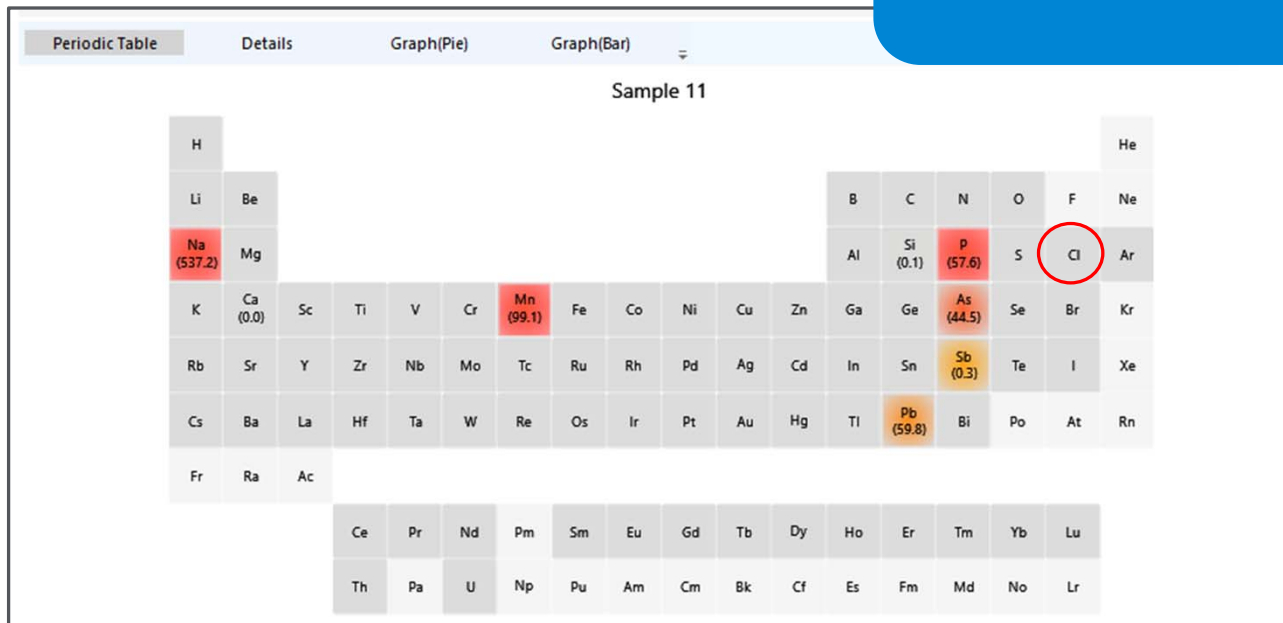
## IntelliQuant 2.0 features to help reduce re-measurement

Full spectrum scan shows everything that is in the sample



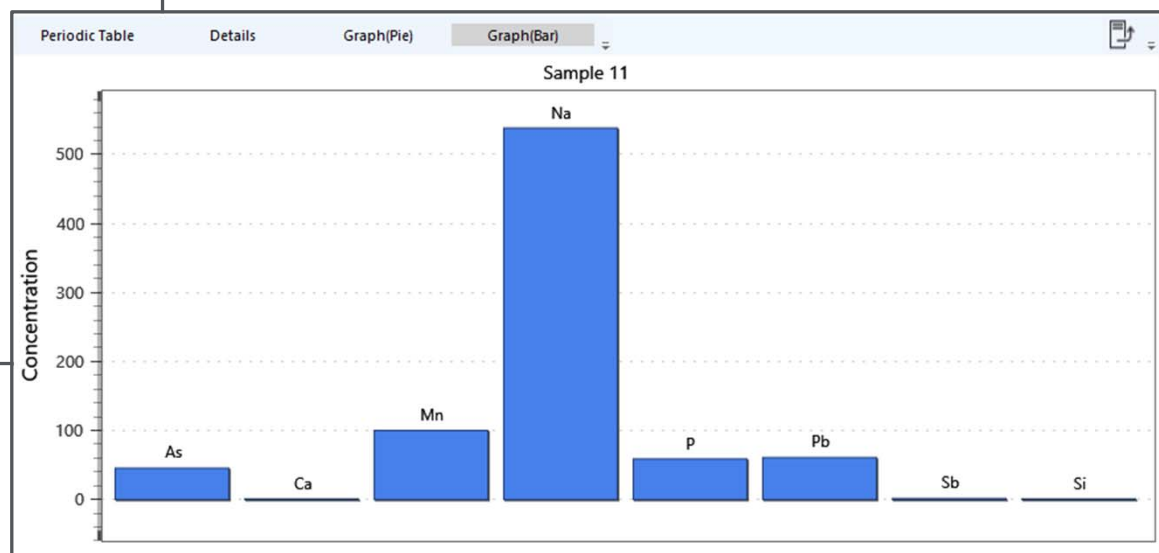
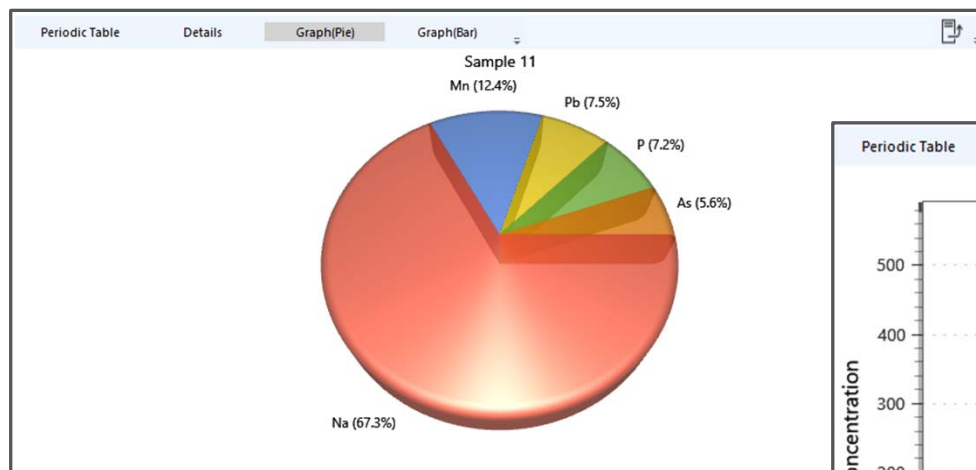
# IntelliQuant 2.0 features to help reduce re-measurement

Periodic table heat map with semi-quant values to quickly identify potential spectral interferences and sample preparation mistakes.



# IntelliQuant 2.0 features to help reduce re-measurement

Smart view to identify composition of a sample



# IntelliQuant 2.0 features to help with method development

Element	Used	Flags	Wavelength	Rating	Concentration	Intensity	Background
Cr	✓		267.716	★★★★	0.01	713.5	1797.4
Eu	✓		397.197	★★★★★	19.34	4432902.5	41958.3
Fe	✓		238.204	★★★★	0.01	940.7	1012.0
Gd	✓		336.224	★★★★★	19.02	687983.0	13457.1
Ho	✓		329.896	★★★★★	20.57	699214.0	19081.5
La	✓		385.852	★★★★★	18.96	1821827.8	23847.6
Mg	✓		279.553	★★★★	-0.01	1221.1	1226.6

List of recommended wavelengths based on standard IntelliQuant analysis or IntelliQuant screening

Element	Used	Flags	Wavelength	Rating	Concentration	Intensity	Background
Gd	✓		238.204	★★★★	0.01	940.7	1012.0
			255.940	★★★★	0.00	87.3	839.0
			342.246	★★★★★	23.72	1481570.6	19501.4
			336.948	★★★★★	21.07	1103125.7	19640.2
Ho	✓		329.896	★★★★★	19.02	687983.0	13457.1
			345.400	★★★★★	25.12	583463.7	12449.7
			339.896	★★★★★	20.57	981837.7	17631.1
			341.644	★★★★★	11.07	57904.7	1690.8
			340.434	★★★★★	19.03	67062.7	2354.1

Analyte: Gd(335.048)  
Confidence: very weak  
Interference: Ho(335.048)  
Confidence: very strong

Star rating system for analyte wavelengths along with question marks next to concentration outliers or those with spectral interferences



Background-corrected scans at multiple wavelengths to confirm presence or absence

## IntelliQuant 2.0 features to help with method development

List of recommended wavelengths based on standard IntelliQuant analysis or IntelliQuant screening

Element	Used	Flags	Wavelength	Rating	Concentration	Intensity	Background
Cr	✓		267.716	★★★★	0.01	713.5	1797.4
Eu	✓		397.197	★★★★★	19.34	4432902.5	41958.3
Fe	✓		238.204	★★★★	0.01	940.7	1012.0
Gd	✓		336.224	★★★★★	19.02	687983.0	13457.1
Ho	✓		339.895	★★★★★	20.57	699214.0	19081.5
La	✓		398.852	★★★★★	18.96	1821827.8	23847.6
Mg	✓		279.553	★★★	-0.01	1221.1	1225.6
Ni							



## IntelliQuant 2.0 features to help with method development

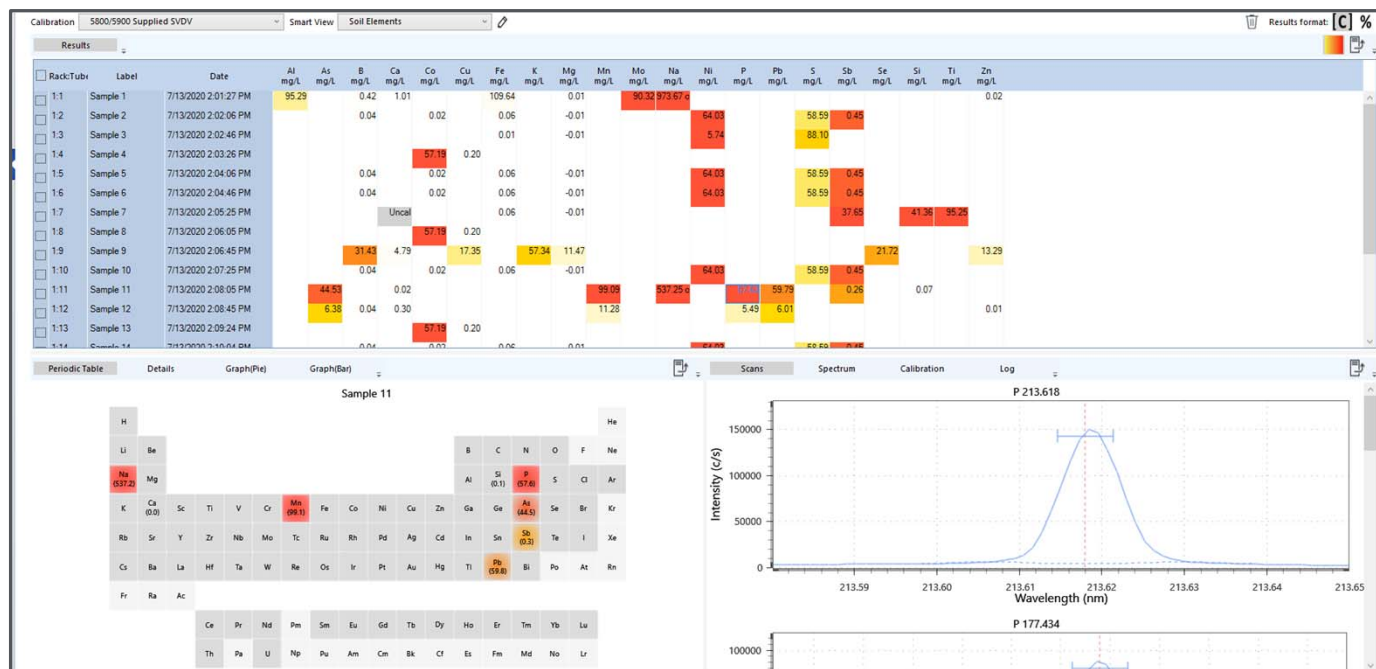
Star rating system for analyte wavelengths along with question marks next to concentration outliers or those with spectral interferences

Element	Used	Flags	Wavelength	Rating	Concentration	Intensity	Background
Gd	✓		238.204	★★★★	0.01	940.7	1012.0
			259.940	*	0.00	87.3	839.0
			342.246	★★★★★	23.72	1461570.6	19501.4
			335.048	* ?	21.07	1103125.7	19640.2
	✓				19.02	687983.0	13457.1
Ho					20.57	583463.7	12449.7
					25.12	981837.7	17631.1
					11.07	57904.7	1690.8
					15.03	67062.7	2354.1
			345.600	* ?	13.44	429784.4	7680.3
	✓		339.895	★★★★★	20.57	699214.0	19081.5
		341.644	★★★★★	20.80	626566.0	28783.0	
		348.484	★★★★★	21.42	535862.0	12040.4	

Analyte: Gd(335.048)  
Confidence: very weak  
Interference: Ho(335.049)  
Confidence: very strong

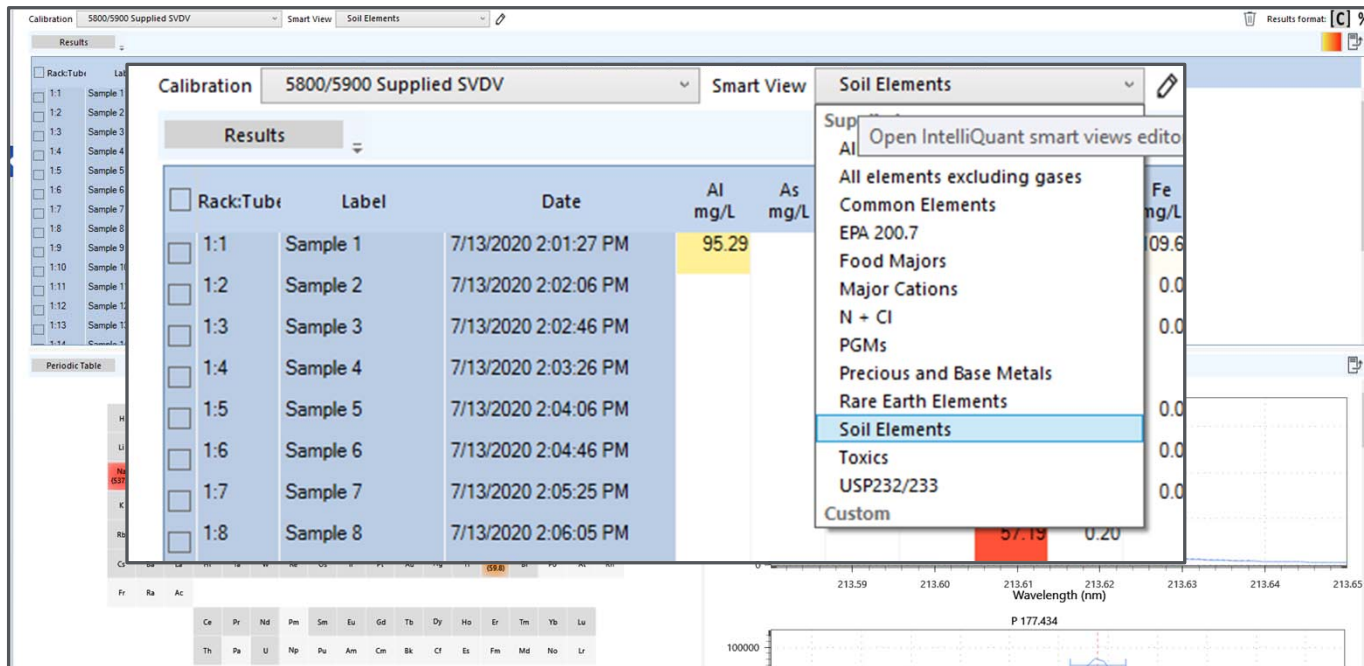
# IntelliQuant 2.0 features to help with method development

Background-corrected scans at multiple wavelengths to confirm presence or absence



# IntelliQuant 2.0 features to help with method development

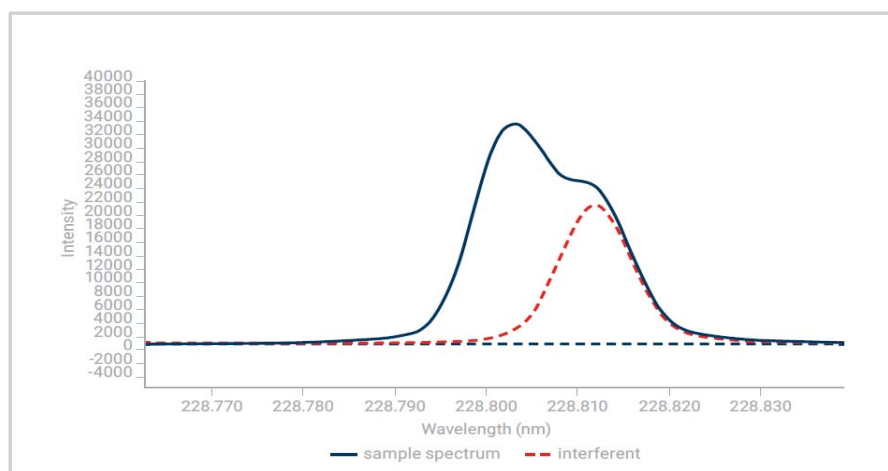
Background-corrected scans at multiple wavelengths to confirm presence or absence



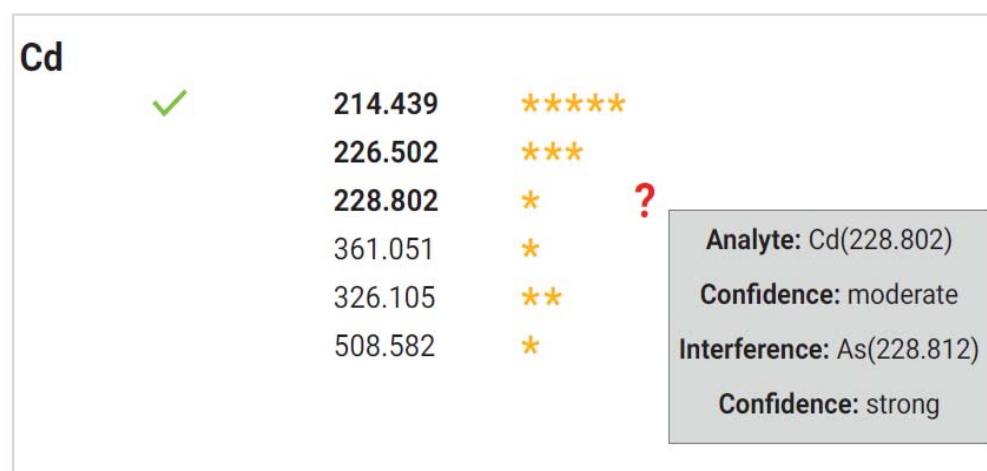
- Customizable element lists
- Customizable calibrations

## How does IntelliQuant 2.0 reduce your remeasurements?

Get greater **sample insight** by automatically detecting spectral overlap.



Cd 228.802nm has unusual peak shape. User is unsure about this result. (User will not see red interferent spectrum, this was added in to help understand existence of overlap)



IntelliQuant automatically identifies Cd 228 had a spectral overlap and ranked it low with one star. IntelliQuant identified that Cd 214.439nm was the best wavelength to report a result with five stars. Data analytics is used to rank the wavelengths.

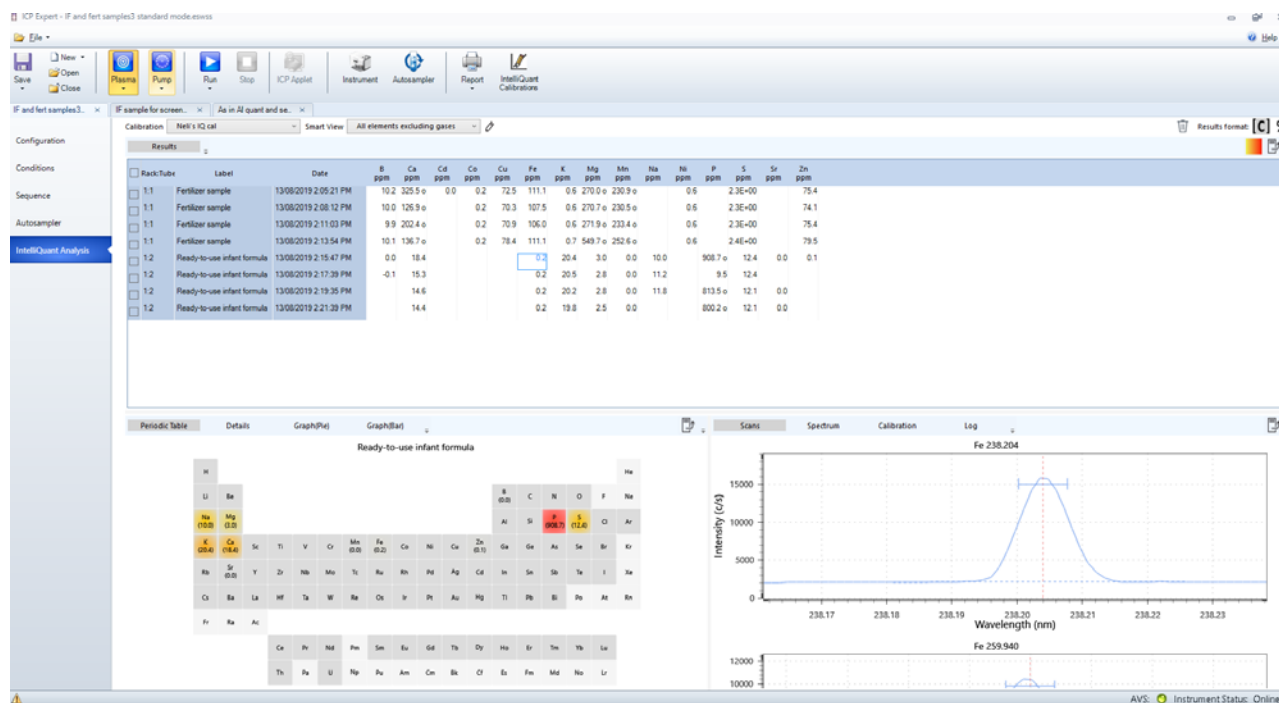
\*Based on a user inserted %RSD threshold in OCF smart tool

# How does IntelliQuant 2.0 reduce your remeasurements?

Have semi quant result in ~20s\*

Know what is in the sample to avoid interference

Know what concentration to make the calibration standards




\*20s achieved using automation

## Real World Examples:

- EPA OTS report estimated that ICP has a 99.9% false positive rate for TI and 25-50% for As
- Similar observations have been made regarding Cd data in soils containing significant amounts of As
- This can lead to expensive and unnecessary remediation and possible exposure to legal action

**Journal of Environmental Quality**



Heavy Metal in the Environment



**A Comparison of Reliability of Soil Cadmium Determination by Standard Spectrometric Methods**

M. B. McBride  
 First published  
 All rights reserved  
 means, electronic  
 retrieval systems  
 Read the full

2011 World of Coal Ash (WOCA) Conference – May 9-12, 2011 in Denver, CO, USA  
<http://www.flyash.info/>

**Evaluation of Inductively Coupled Plasma (ICP) False Positives for Arsenic and Trace Metals Analysis in Coal Ash and Coal Ash Media**

**Richard J. Haley & Alder Laboratories,**

LATEST ISSUE >  
 Volume 30, Issue 3  
 Summer 2020

**KEYWORDS:**  
 dynamic reaction

**ABSTRACT**

The phenomenon of false positive results in ICP-AES data reported by the U.S. Environmental Protection Agency has been documented in several studies. This phenomenon is caused by the presence of arsenic in the samples.

Research Article

**Arsenic and thallium data in environmental samples: Fact or fiction?**

Susan D. Chapnick, Leonard C. Pitts, Nancy C. Rothman

First published: 16 September 2010 | <https://doi.org/10.1002/rem.20260> | Citations: 3

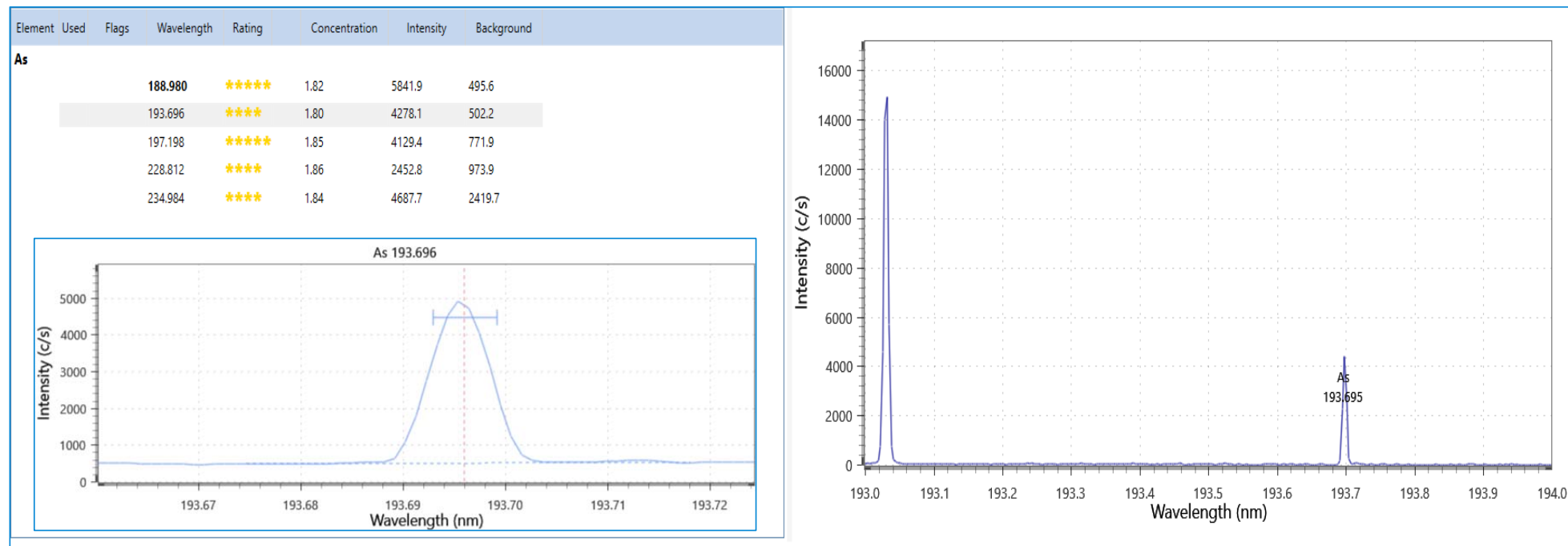
**Abstract**

A U.S. Environmental Protection Agency Office of Technical Standards Alert estimated that environmental data reported using inductively coupled plasma spectrometry (ICP-AES) has a

# Real World Examples

## AI interference on As in earth sample

US EPA method 6010 recommends the As193.696 nm emission line



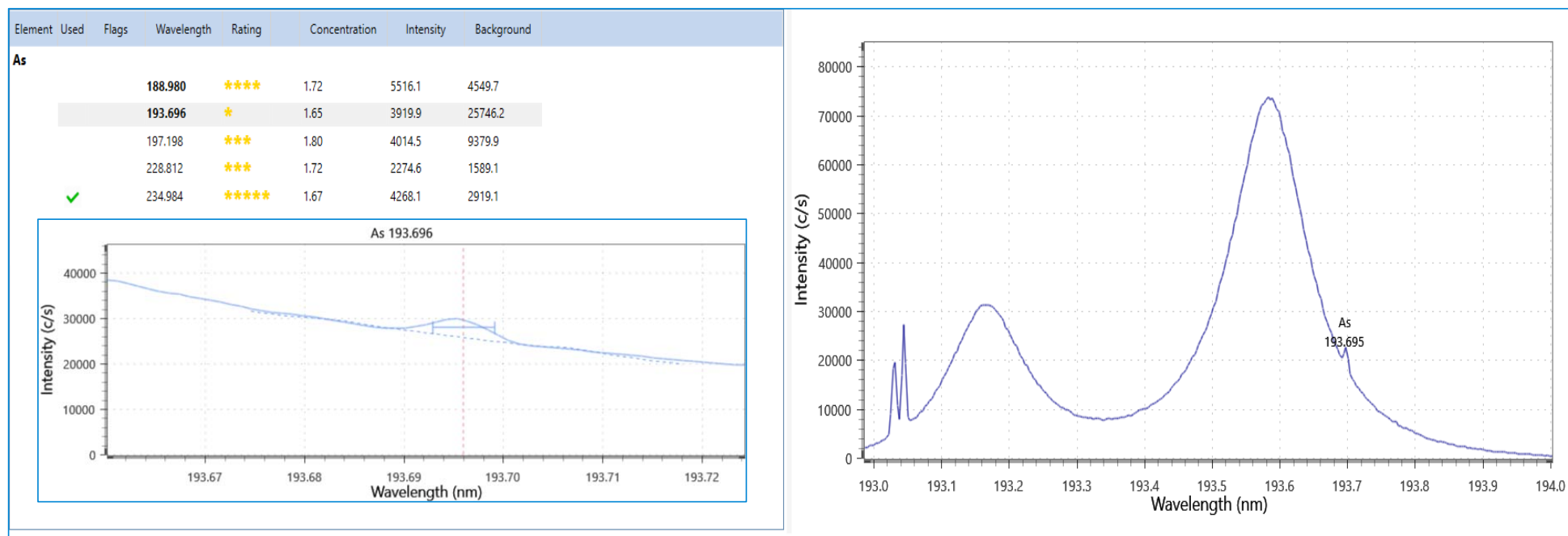
2 mg/L As in 0ppm Al (minimal background signal). Carbon emission line at 193.028 nm

INSET – As wavelength scan at 193.696 nm

# Real World Examples

## Al interference on As in earth sample

US EPA method 6010 recommends the As193.696 nm emission line



2 mg/L As in 5,000ppm Al (broad aluminum auto-ionizing doublet is clearly visible)

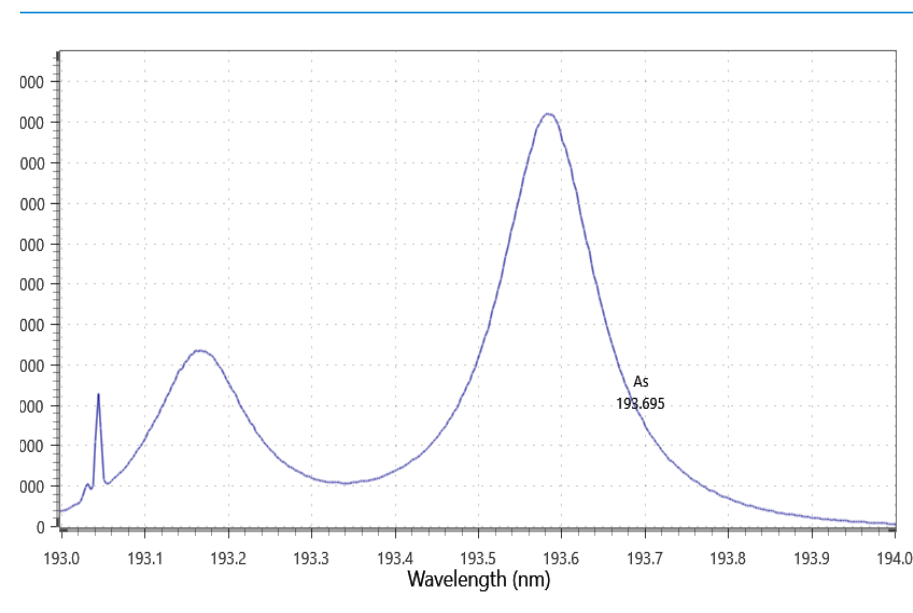
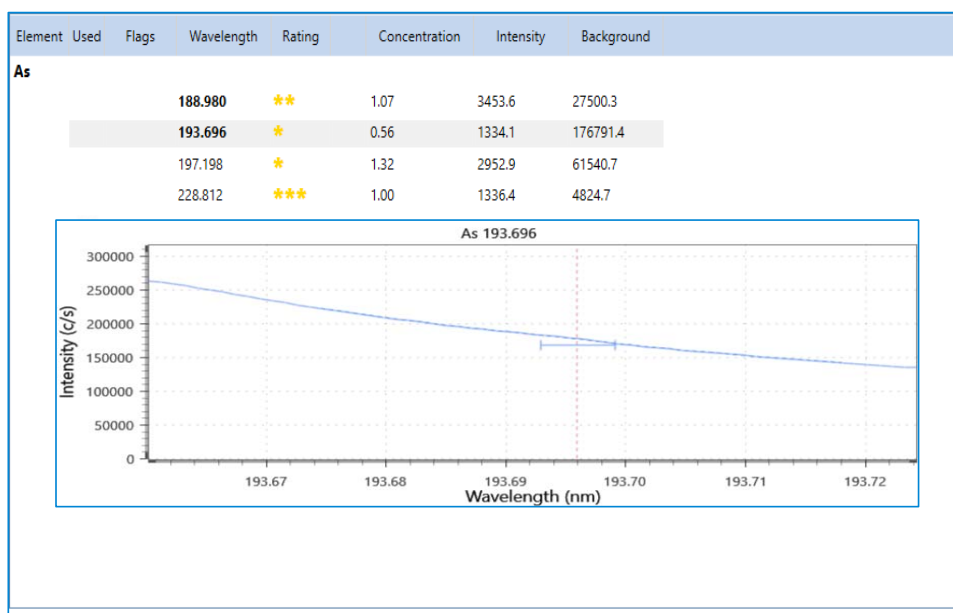
INSET – As wavelength scan at 193.696 nm



# Real World Examples

## AI interference on As in earth sample

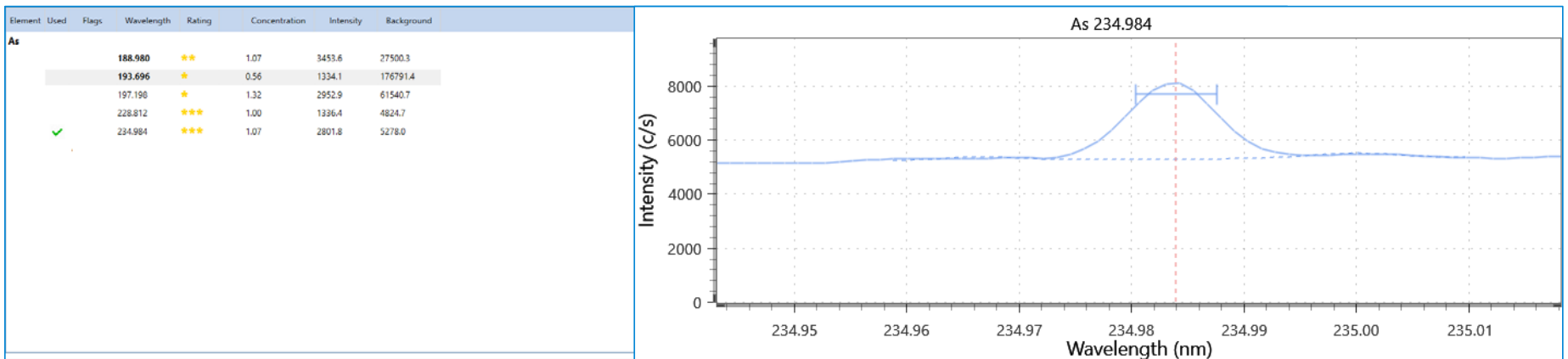
US EPA method 6010 recommends the As193.696 nm emission line



2 mg/L As in 50,000ppm Al  
INSET – As wavelength scan at 193.696 nm

# The Solution: IntelliQuant for High Aluminium on As

How do I recognize when AI is affecting my results?



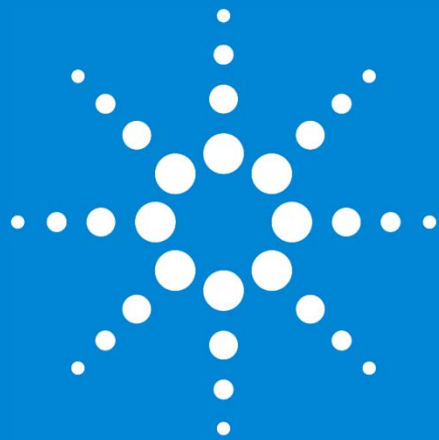
2 mg/L As in 50,000ppm Al

IntelliQuant analyzes a suite of As lines for SRBR and recommends As 234.984nm line in the presence of high Al.

IntelliQuant informs the operator that the result from the As 193 line is of poor quality for this particular sample and recommends a suitable alternative wavelength with a lower MDL.

Thank you for your attention!  
Questions?





# Agilent

Trusted Answers