

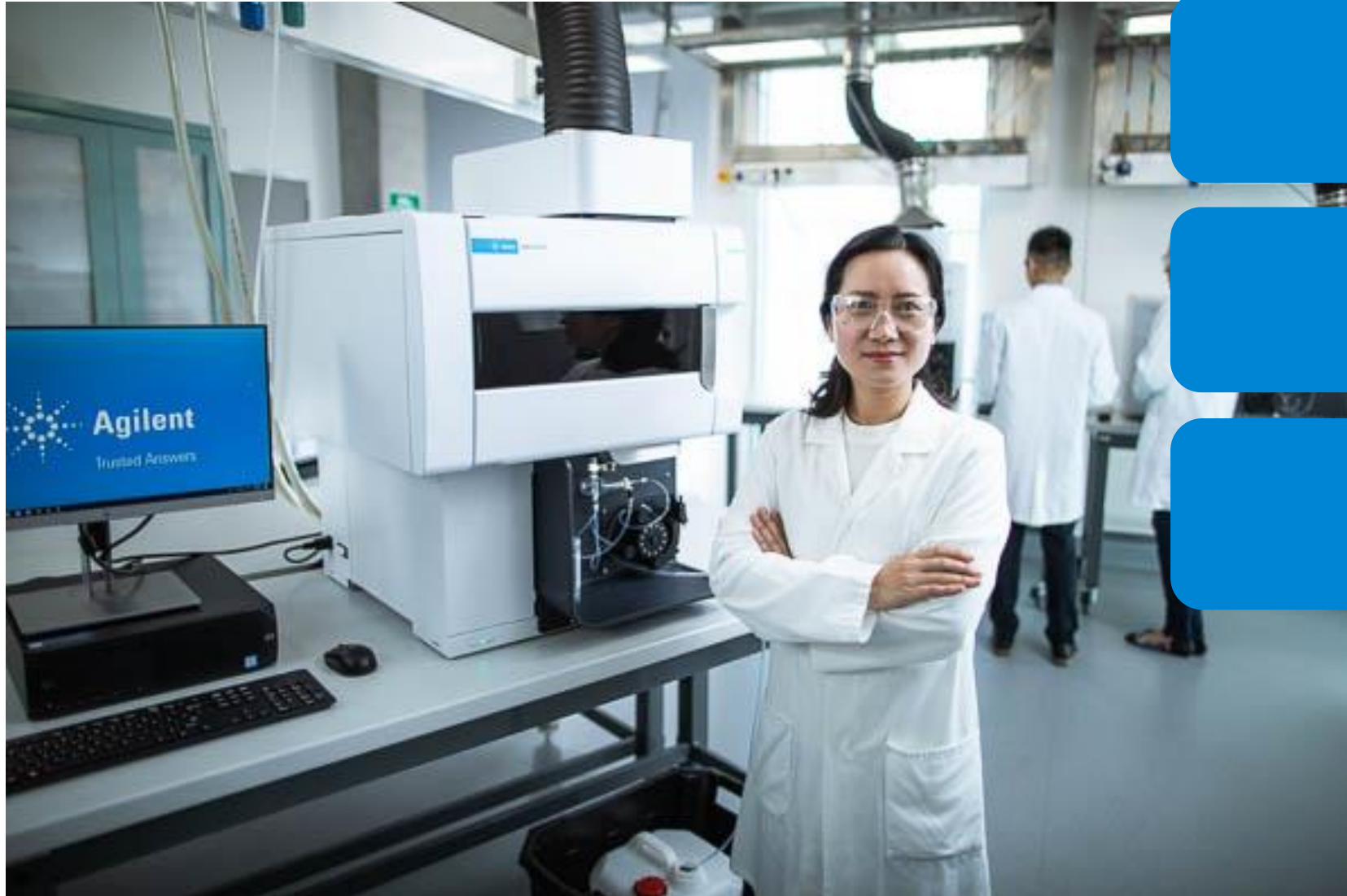
# Streamlining Method Development for Metals Analysis Using Agilent 5900 ICPOES and IntelliQuant 2.0

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Metals Analysis and Remediation Session  
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# Agilent Smart Tools reduce your remeasurement



Intelligent

Efficient

Confident

# 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

# What issues lead to sample remeasurement?

## Sample Related

- Spectral Interference
- Analytical Line Saturated
- Improper prep

## Operation Related

- Nebulizer blockage
- Peri pump tubing disconnected
- Internal Standard failure
- Analyte carry-over



# 5800/5900 ICP-OES with 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

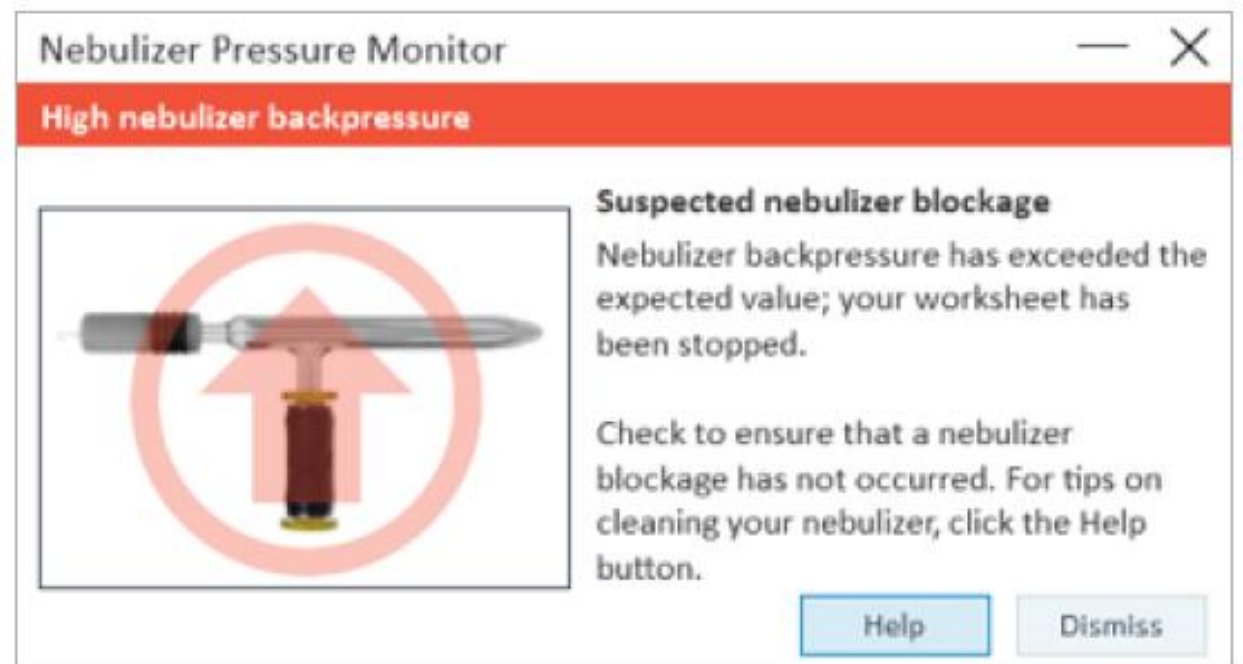
# Smart tools for operational insight

 Instrument Options

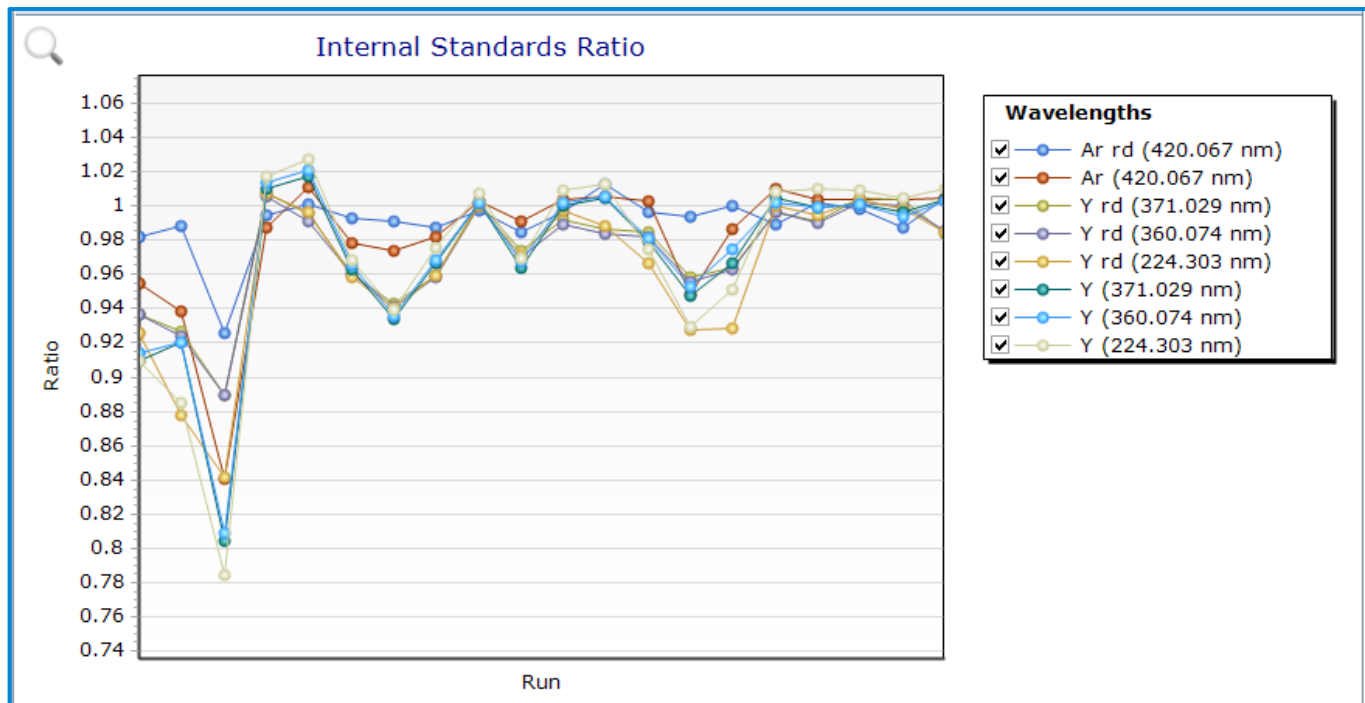
**Instrument Options**

- ☐ Enable polychromator boost purge on connection
- ☐ Plasma auto-extinguish idle timeout (mins):
- ☒ Enable Neb Alert (kPa) 
- Pressure lower limit:
- Pressure upper limit:
- ☒ Stop analysis if pressure falls outside limits 
- ☒ Enable reminders on plasma ignition

NebAlert



# Smart tools for operational insight



Internal Standard  
monitor



# Smart tools for operational insight

## Outlier Conditional Formatting (OCF)

|                                     | Rack:Tube | Solution Label | Outlier Summary | Co<br>8.615 nm<br>ppm | Co<br>230.786 nm<br>ppm | Cr<br>267.716 nm<br>ppm | Cu<br>327.395 nm<br>ppm | Fe<br>273.358 nm<br>ppm | K<br>766.491 nm<br>ppm | La<br>408.671 nm<br>ppm | Li<br>670.783 nm<br>ppm | Mn<br>279.80<br>ppm |
|-------------------------------------|-----------|----------------|-----------------|-----------------------|-------------------------|-------------------------|-------------------------|-------------------------|------------------------|-------------------------|-------------------------|---------------------|
| <input type="checkbox"/>            | 2:2       | ORESA 45e 2    |                 | 1.3645                | 1.1683                  | 19.5747                 | 14.8628                 | 3340.6496               | 57.8595                | 0.0167                  | 0.1325                  | 7                   |
| <input checked="" type="checkbox"/> | 2:3       | SRM 2781 1     |                 | 0.2291                | 0.1247                  | 3.7995                  | 11.3385                 | 520.5713                | 93.9258                | 0.3927                  | 0.1407                  | 107                 |
| <input type="checkbox"/>            | 2:4       | SRM 2781 2     |                 | 0.2323                | 0.1270                  | 3.8096                  | 11.8384                 | 531.5788                | 97.1176                | 0.3894                  | 0.1445                  | 109                 |
| <input type="checkbox"/>            | 2:5       | SRM 2782 1     | A               | 1.5177                | 1.4996                  | 2.3091                  | 53.5224                 | 5852.2210 o             | 71.4071                | 1.2246                  | 0.1355                  | 55                  |
| <input type="checkbox"/>            | 2:6       | SRM 2782 2     |                 | 1.4482                | 1.4303                  | 2.1963                  | 51.4172                 | 5474.6380               | 67.6546                | 1.1696                  | 0.1378                  | 52                  |
| <input type="checkbox"/>            | 2:1       | ORESA 45e 1    |                 | 1.4282                | 1.2215                  | 20.2531                 | 15.9087                 | 3385.5674               | 61.0864                | 0.0047                  |                         |                     |
| <input type="checkbox"/>            | 2:2       | ORESA 45e 2    |                 | 1.4042                | 1.1999                  | 20.0833                 | 15.1864                 | 3409.9162               | 59.4210                | 0.0166                  |                         |                     |
| <input type="checkbox"/>            | 2:3       | SRM 2781 1     |                 | 0.2293                | 0.1244                  | 3.8020                  | 11.4742                 | 534.8713                | 96.1179                | 0.3911                  | 0.1418                  | 110                 |
| <input type="checkbox"/>            | 2:4       | SRM 2781 2     |                 | 0.2405                | 0.1306                  | 3.9745                  | 12.0563                 | 550.3115                | 100.0555               | 0.4072                  | 0.1418                  | 112                 |
| <input type="checkbox"/>            | 2:5       | SRM 2782 1     | A               | 1.5041                | 1.4722                  | 2.2825                  | 53.6837                 | 5822.7552 o             | 71.1665                | 1.2091                  | 0.1284                  | 55                  |
| <input type="checkbox"/>            | 2:6       | SRM 2782 2     |                 | 1.4680                | 1.4463                  | 2.2234                  | 51.6004                 | 5436.4548               | 66.9066                | 1.1841                  | 0.1276                  | 51                  |
| <input type="checkbox"/>            | 2:1       | ORESA 45e 1    |                 | 1.4573                | 1.2448                  | 20.6513                 | 16.0510                 | 3447.9631               | 62.1334                | 0.0053                  | 0.1377                  | 4                   |
| <input type="checkbox"/>            | 2:2       | ORESA 45e 2    |                 | 1.4048                | 1.2037                  | 20.1358                 | 15.2630                 | 3419.2366               | 59.4457                | 0.0166                  | 0.1269                  | 7                   |
| <input type="checkbox"/>            | 2:3       | SRM 2781 1     |                 | 0.2334                | 0.1267                  | 3.8933                  | 11.6111                 | 551.2126                | 98.6493                | 0.3990                  | 0.1374                  | 113                 |

|                          | Rack:Tube | Solution Label | Outlier Summary | Co<br>8.615 nm<br>ppm | Co<br>230.786 nm<br>ppm | Cr<br>267.716 nm<br>ppm | Cu<br>327.395 nm<br>ppm | Fe<br>273.358 nm<br>ppm | K<br>766.491 nm<br>ppm | La<br>408.671 nm<br>ppm | Li<br>670.783 nm<br>ppm | Mn<br>279.80<br>ppm |
|--------------------------|-----------|----------------|-----------------|-----------------------|-------------------------|-------------------------|-------------------------|-------------------------|------------------------|-------------------------|-------------------------|---------------------|
| <input type="checkbox"/> | 2:2       | ORESA 45e 2    |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:3       | SRM 2781 1     |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:4       | SRM 2781 2     |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:5       | SRM 2782 1     | A               |                       |                         |                         |                         | 5852.2210 o             |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:6       | SRM 2782 2     |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:1       | ORESA 45e 1    |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:2       | ORESA 45e 2    |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:3       | SRM 2781 1     |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:4       | SRM 2781 2     |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:5       | SRM 2782 1     | A               |                       |                         |                         |                         | 5822.7552 o             |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:6       | SRM 2782 2     |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:1       | ORESA 45e 1    |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:2       | ORESA 45e 2    |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |
| <input type="checkbox"/> | 2:3       | SRM 2781 1     |                 |                       |                         |                         |                         |                         |                        |                         |                         |                     |

☒ Display flagged only

| Flag | Flag Rule                                           | Pass Value | Enable                              |                                                              |
|------|-----------------------------------------------------|------------|-------------------------------------|--------------------------------------------------------------|
| A    | Result Concentration Overage                        | N/A        | <input checked="" type="checkbox"/> |                                                              |
| B    | Result Concentration % RSD > Pass Value             | 10.00      | <input type="checkbox"/>            | <input type="checkbox"/> Only apply when result > 10.0 * MDL |
| C    | Result Concentration < MDL                          | N/A        | <input type="checkbox"/>            |                                                              |
| D    | Internal standard % recovery variation > Pass Value | 15.00      | <input type="checkbox"/>            |                                                              |
| E    | QC Equation Test Fail                               | N/A        | <input type="checkbox"/>            |                                                              |



# Smart tools for operational insight

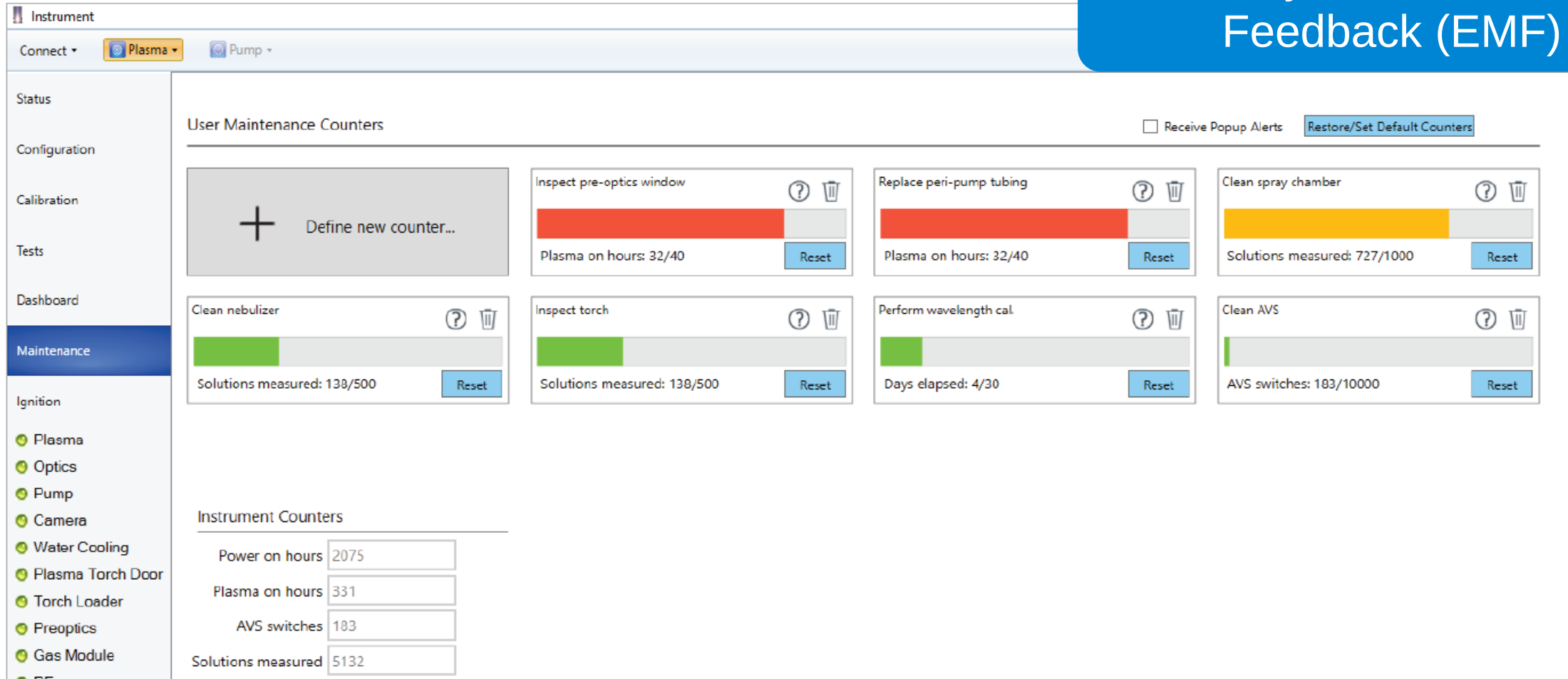
## Outlier Conditional Formatting (OCF)

☒ Display flagged only

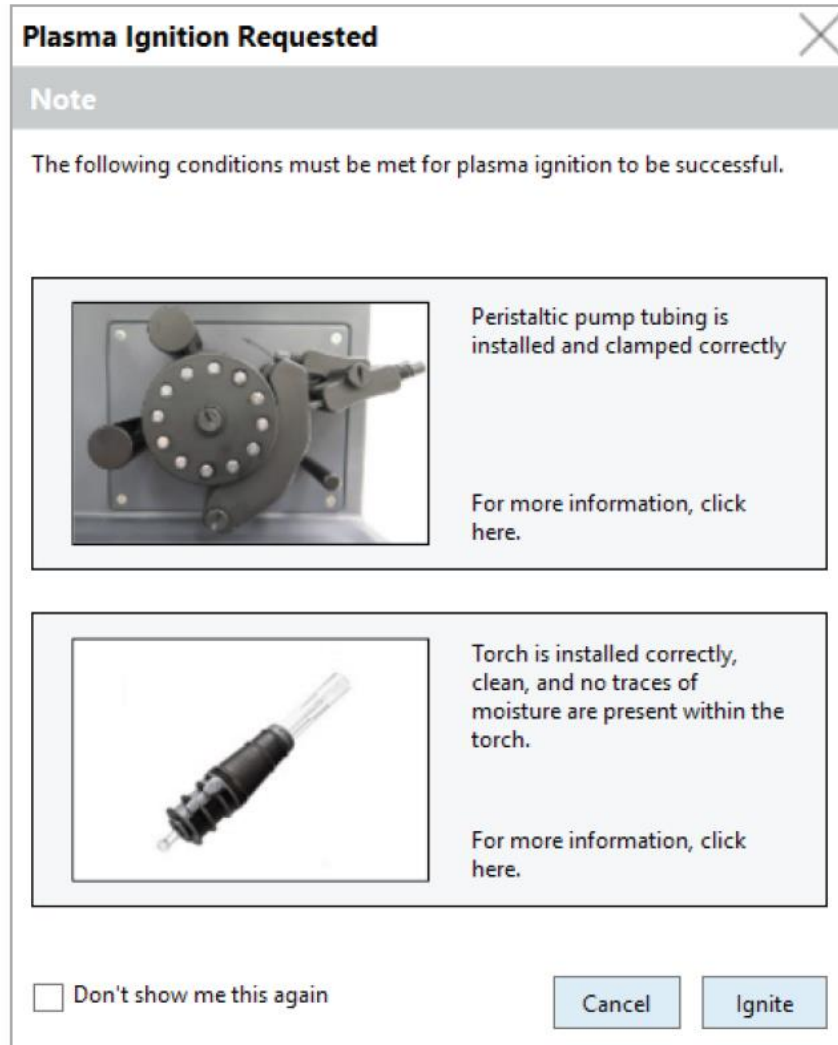
| Flag | Flag Rule                                           | Pass Value | Enable                              |                                                              |
|------|-----------------------------------------------------|------------|-------------------------------------|--------------------------------------------------------------|
| A    | Result Concentration Overage                        | N/A        | <input checked="" type="checkbox"/> |                                                              |
| B    | Result Concentration % RSD > Pass Value             | 10.00      | <input type="checkbox"/>            | <input type="checkbox"/> Only apply when result > 10.0 * MDL |
| C    | Result Concentration < MDL                          | N/A        | <input type="checkbox"/>            |                                                              |
| D    | Internal standard % recovery variation > Pass Value | 15.00      | <input type="checkbox"/>            |                                                              |
| E    | QC Equation Test Fail                               | N/A        | <input type="checkbox"/>            |                                                              |

# Smart tools for operational insight

## Early Maintenance Feedback (EMF)



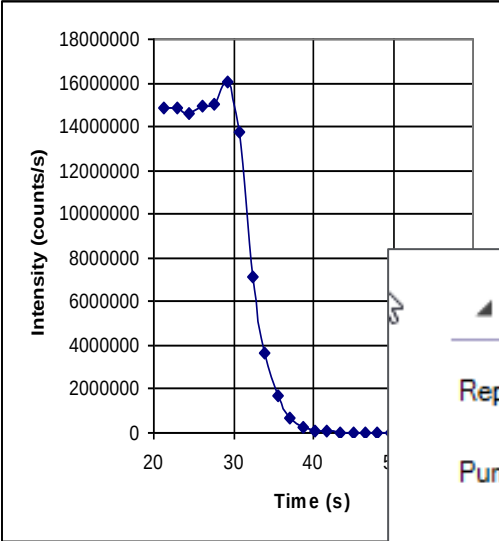
# Smart tools for operational insight



## Optimized plasma ignition

- Prevents difficult ignition
- Prevents torch melts
- Prevents torch 'fountains'

# Smart tools for operational insight



## Intelligent Rinse

### Common Conditions

Replicates:  ⓘ

Pump speed (rpm):  ⓘ

Uptake delay (s):  ⓘ ☒ Fast Pump

Max rinse time (s):  ⓘ ☒ Fast Pump

Enable Intelligent Rinse ☒ 

Re-acquire baseline on timeout ☐

| Intelligent Rinse                   | Intelligent Rinse Method |
|-------------------------------------|--------------------------|
| <input checked="" type="checkbox"/> | Moderate                 |
| <input checked="" type="checkbox"/> | Thorough                 |
| <input checked="" type="checkbox"/> | Moderate                 |
| <input checked="" type="checkbox"/> | Quick                    |
| <input checked="" type="checkbox"/> | Moderate                 |

# What issues lead to sample remeasurement?

## Sample Related

- Spectral Interference
- Analytical Line Saturated
- Improper prep

## Operation Related

- Nebulizer blockage
- Peri pump tubing disconnected
- Internal Standard failure
- Analyte carry-over



# Smart tools that reduce your remeasurement

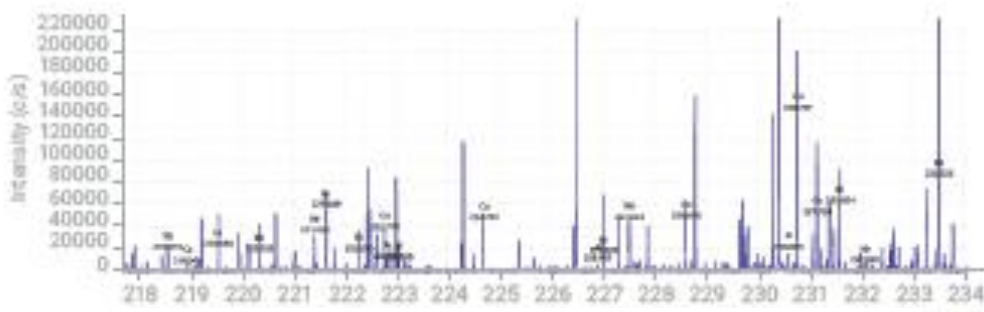
Use Smart Tools to gain **insights into your samples**

- 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

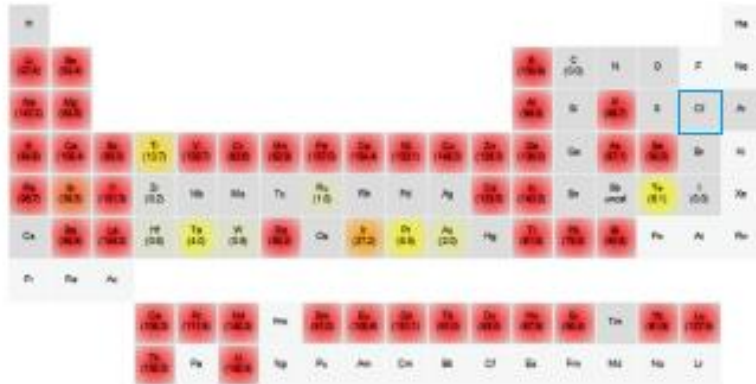
Smart Tools

Sample Insights

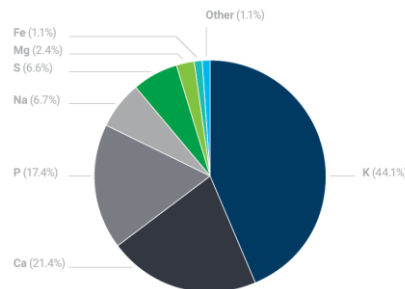
# IntelliQuant 2.0 features to help reduce re-measurement



Full spectrum scan shows everything that is in the sample



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

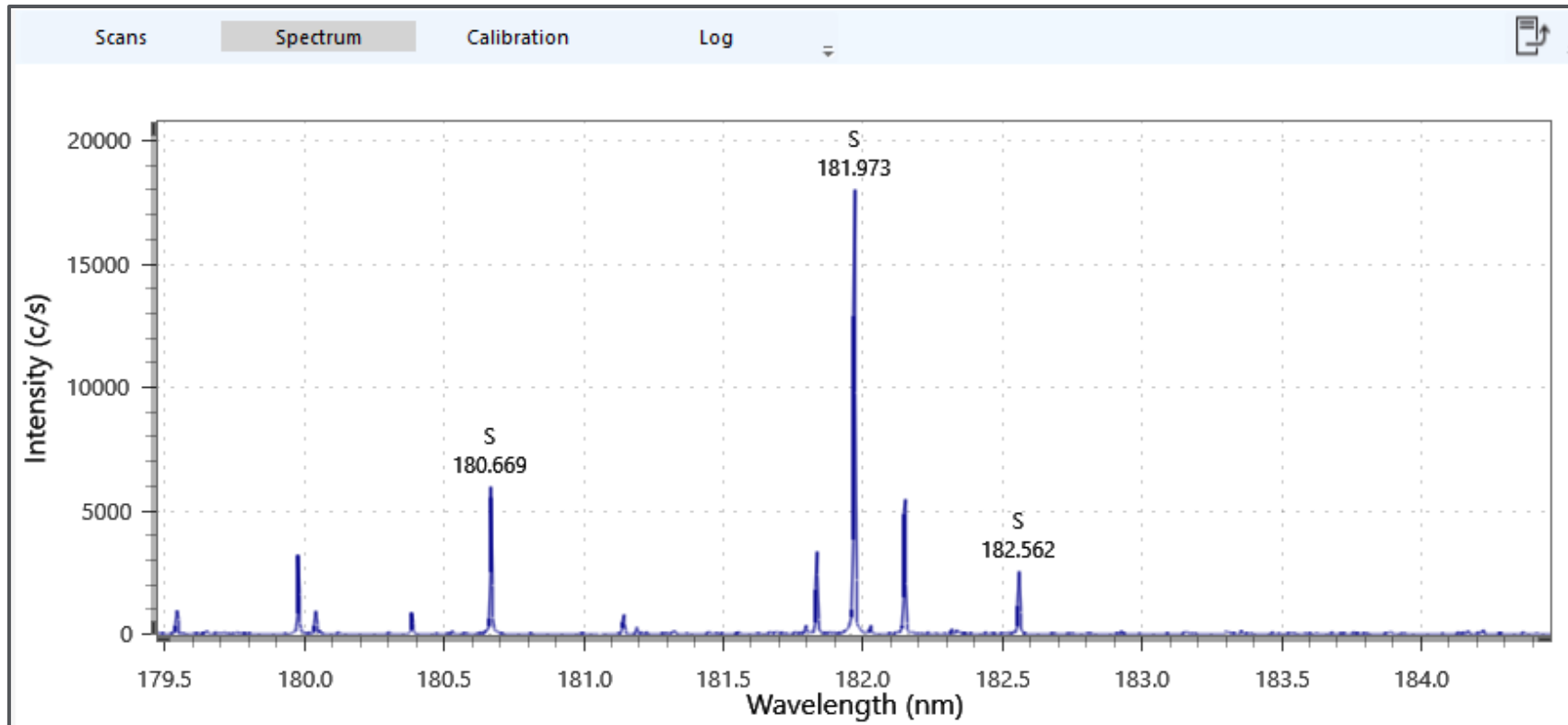


Smart view to identify composition of a sample



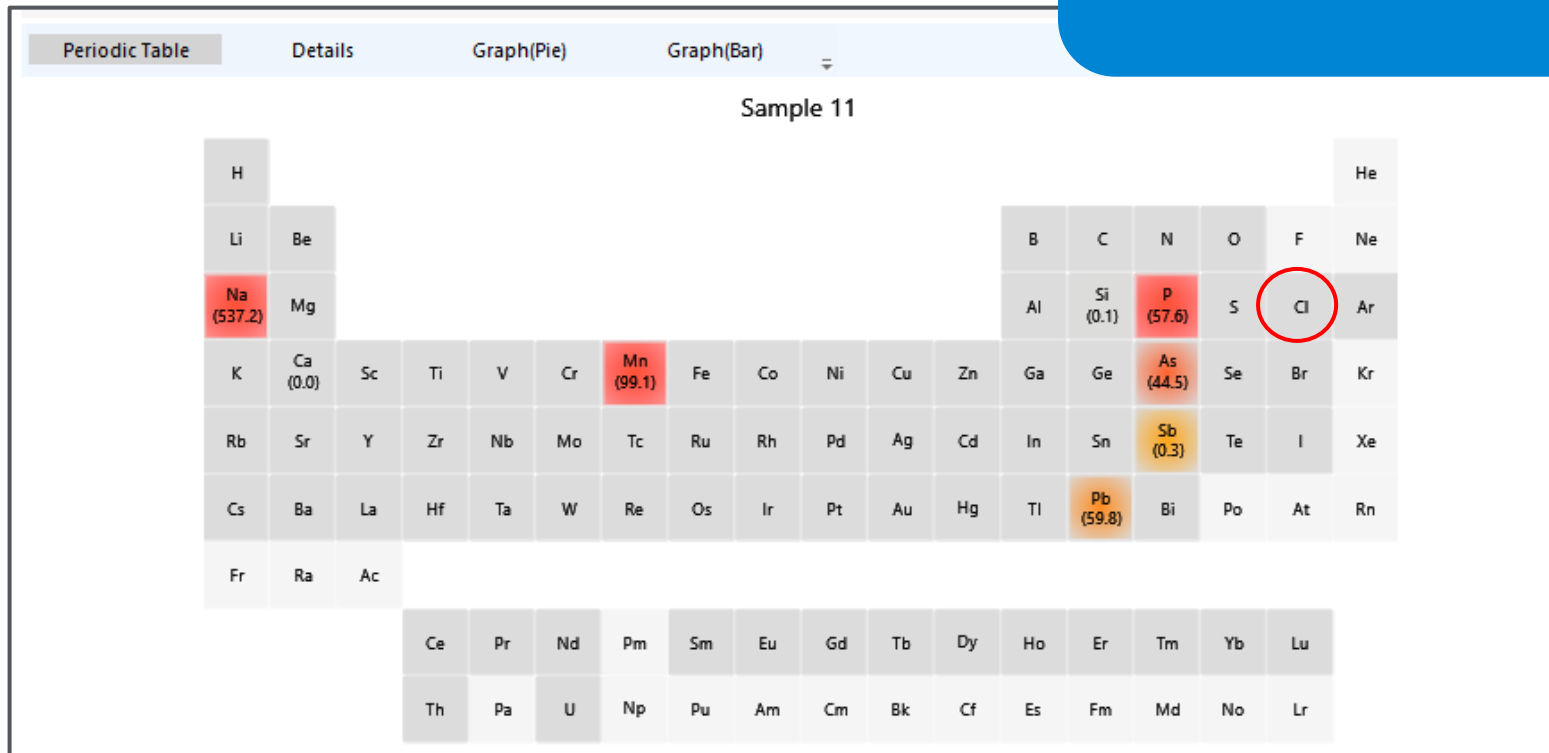
# IntelliQuant 2.0 features to help reduce re-measurement

Full spectrum scan shows everything that is in the sample



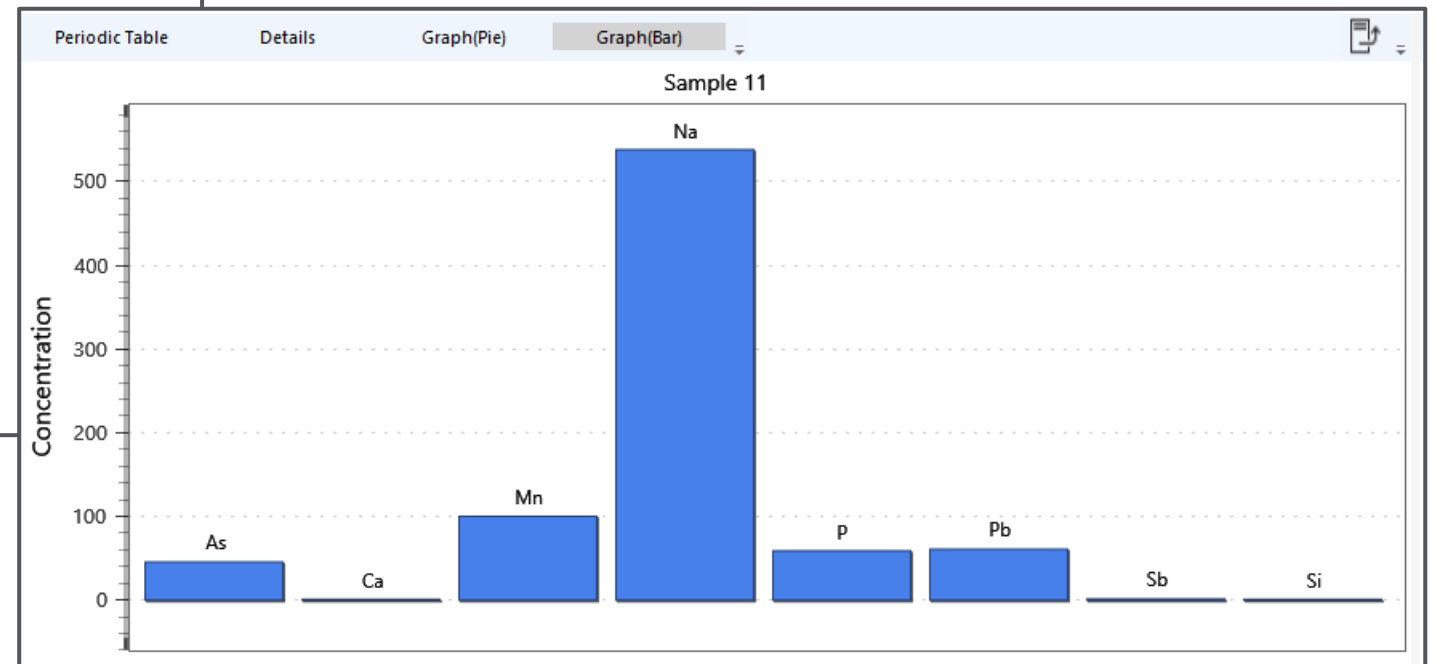
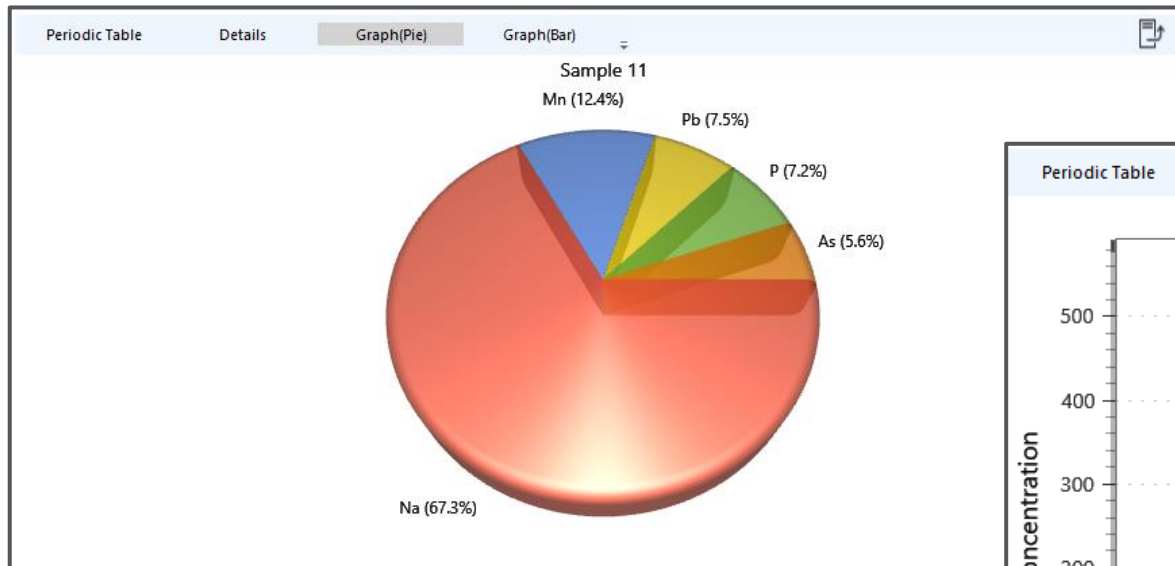
# IntelliQuant 2.0 features to help reduce re-measurement

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

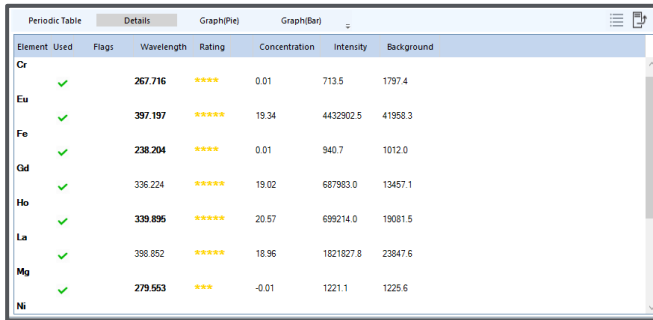


# IntelliQuant 2.0 features to help reduce re-measurement

Smart view to identify composition of a sample

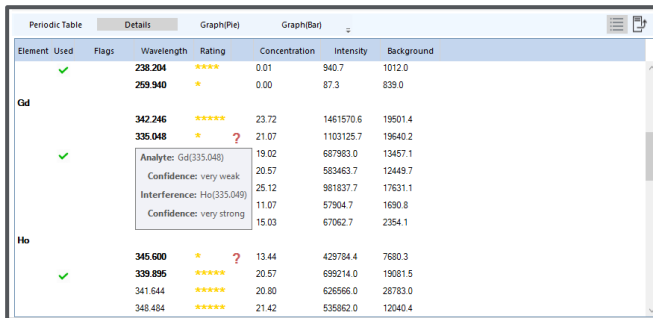


# IntelliQuant 2.0 features to help with method development



| 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      | ✓    |       | 339.896    | ★★★★   | 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      |      |       |            |        |               |           |            |

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     |
|         |      |       | 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    |
|         |      |       |            |        | 20.57         | 583463.7  | 12449.7    |
|         |      |       |            |        | 25.12         | 981837.7  | 17631.1    |
|         |      |       |            |        | 11.07         | 57904.7   | 1690.8     |
|         |      |       |            |        | 15.03         | 67062.7   | 2354.1     |
| Ho      | ✓    |       | 345.600    | *      | 13.44         | 429784.4  | 7680.3     |
|         |      |       | 339.896    | ★★★★   | 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

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

| Periodic Table |      | Details | Graph(Pie) | Graph(Bar) |               |           |            |  |
|----------------|------|---------|------------|------------|---------------|-----------|------------|--|
| 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             |      |         |            |            |               |           |            |  |

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

# IntelliQuant 2.0 features to help with method development

| <div> Periodic Table Details Graph(Pie) Graph(Bar) </div> |      |       |                                                                                                           |        |               |           |            |  |
|-----------------------------------------------------------|------|-------|-----------------------------------------------------------------------------------------------------------|--------|---------------|-----------|------------|--|
| 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    |  |
|                                                           | ✓    |       | <div> Analyte: Gd(335.048) Confidence: very weak Interference: Ho(335.049) Confidence: very strong </div> |        | 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    |  |

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

# IntelliQuant 2.0 features to help with method development



# IntelliQuant 2.0 features to help with method development

| Calibration              |           | 5800/5900 Supplied SVDV |                      | Smart View |            | Soil Elements |  |
|--------------------------|-----------|-------------------------|----------------------|------------|------------|---------------|--|
| Results                  |           |                         |                      |            |            |               |  |
| <input type="checkbox"/> | Rack:Tube | Label                   | Date                 | Al<br>mg/L | As<br>mg/L | Fe<br>mg/L    |  |
| <input type="checkbox"/> | 1:1       | Sample 1                | 7/13/2020 2:01:27 PM | 95.29      |            | 09.6          |  |
| <input type="checkbox"/> | 1:2       | Sample 2                | 7/13/2020 2:02:06 PM |            |            | 0.0           |  |
| <input type="checkbox"/> | 1:3       | Sample 3                | 7/13/2020 2:02:46 PM |            |            | 0.0           |  |
| <input type="checkbox"/> | 1:4       | Sample 4                | 7/13/2020 2:03:26 PM |            |            | 0.0           |  |
| <input type="checkbox"/> | 1:5       | Sample 5                | 7/13/2020 2:04:06 PM |            |            | 0.0           |  |
| <input type="checkbox"/> | 1:6       | Sample 6                | 7/13/2020 2:04:46 PM |            |            | 0.0           |  |
| <input type="checkbox"/> | 1:7       | Sample 7                | 7/13/2020 2:05:25 PM |            |            | 0.0           |  |
| <input type="checkbox"/> | 1:8       | Sample 8                | 7/13/2020 2:06:05 PM |            |            | 0.20          |  |

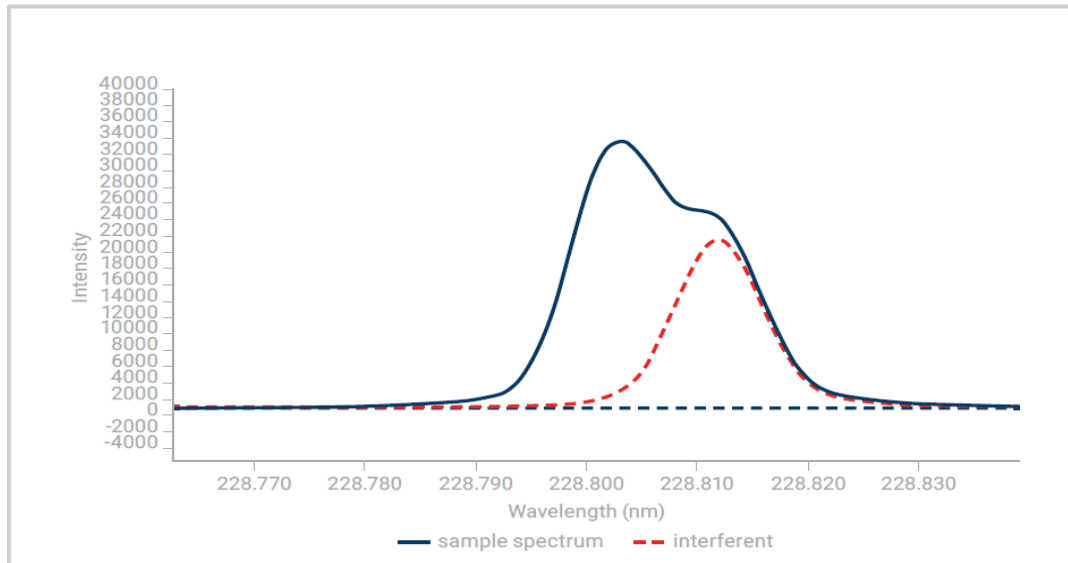
Background-corrected scans at multiple wavelengths to confirm presence or absence

- 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)

|    |   |         |       |   |
|----|---|---------|-------|---|
| Cd | ✓ | 214.439 | ***** |   |
|    |   | 226.502 | ***   |   |
|    |   | 228.802 | *     | ? |
|    |   | 361.051 | *     |   |
|    |   | 326.105 | **    |   |
|    |   | 508.582 | *     |   |

Analyte: Cd(228.802)

Confidence: moderate

Interference: As(228.812)

Confidence: strong

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.

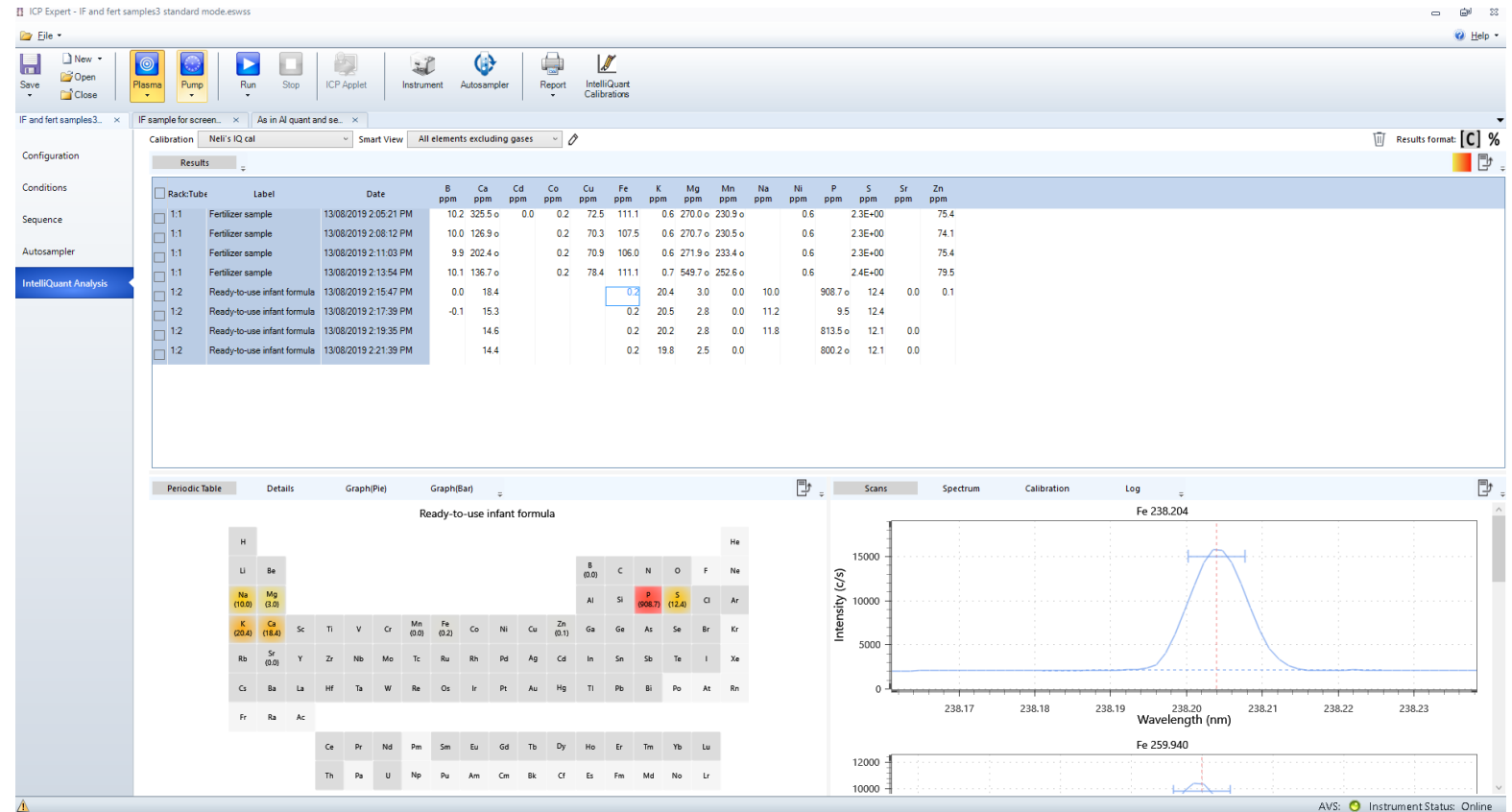
\*Based on a user inserted %RSD threshold in OCF smart tool

# 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 Tl 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

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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

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KEYWORDS: dynamic reaction

#### ABSTRACT

The phenomenon of false positive results in ICP-AES analysis of environmental samples via ICP-AES has been documented. It is a well-known fact that a reliable method of analysis is required for the protection of the public. The U.S. Environmental Protection Agency has reported that this phenomenon is a significant problem in the analysis of environmental samples.

## REMEDATION

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Research Article

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

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

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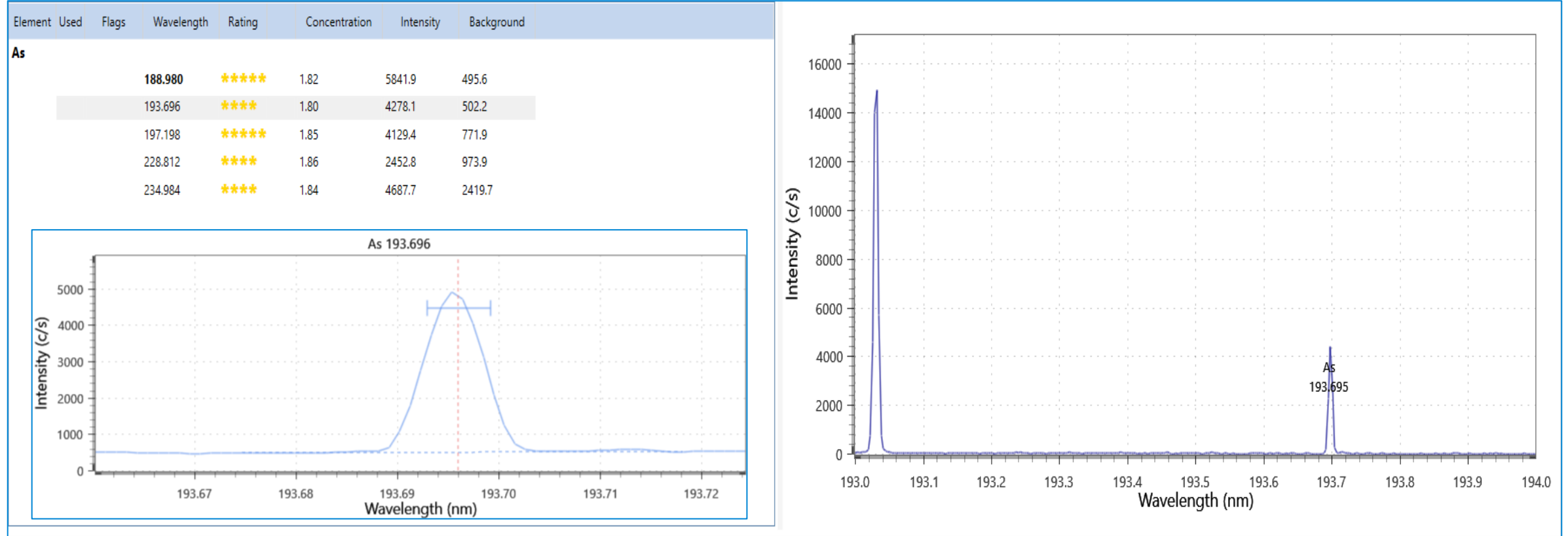
#### Abstract

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

## Al interference on As in earth sample

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



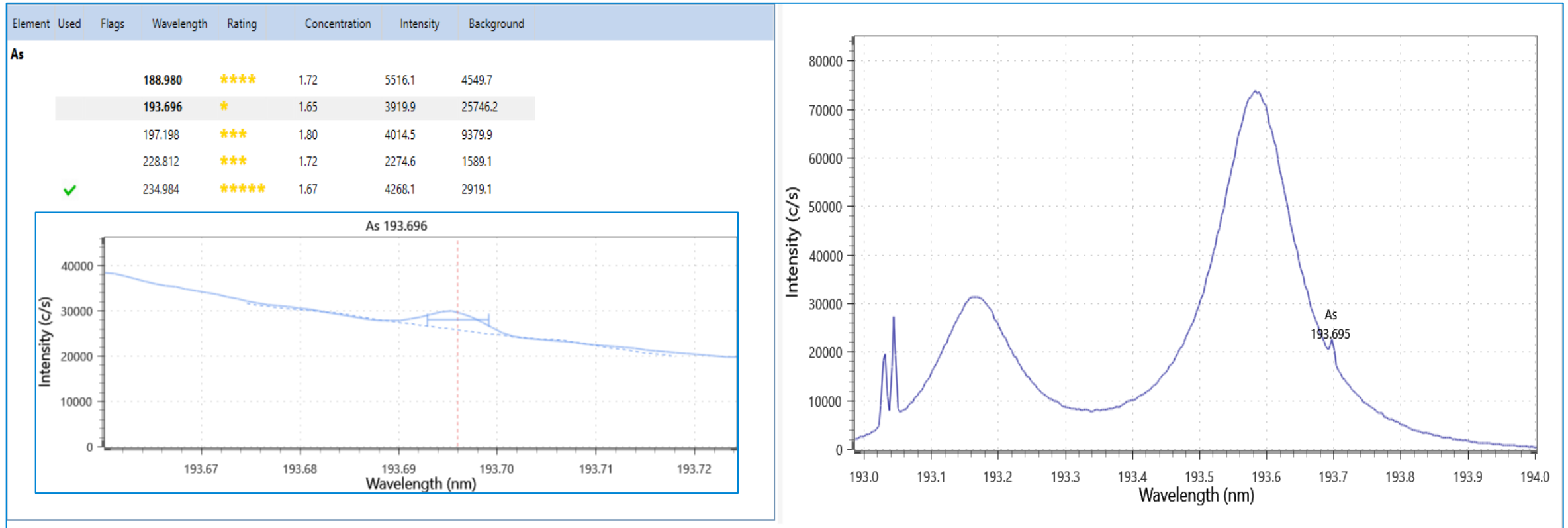
2 mg/L As in 0ppm Al (minimal background signal). Carbon emission line at 193.028 nm)

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



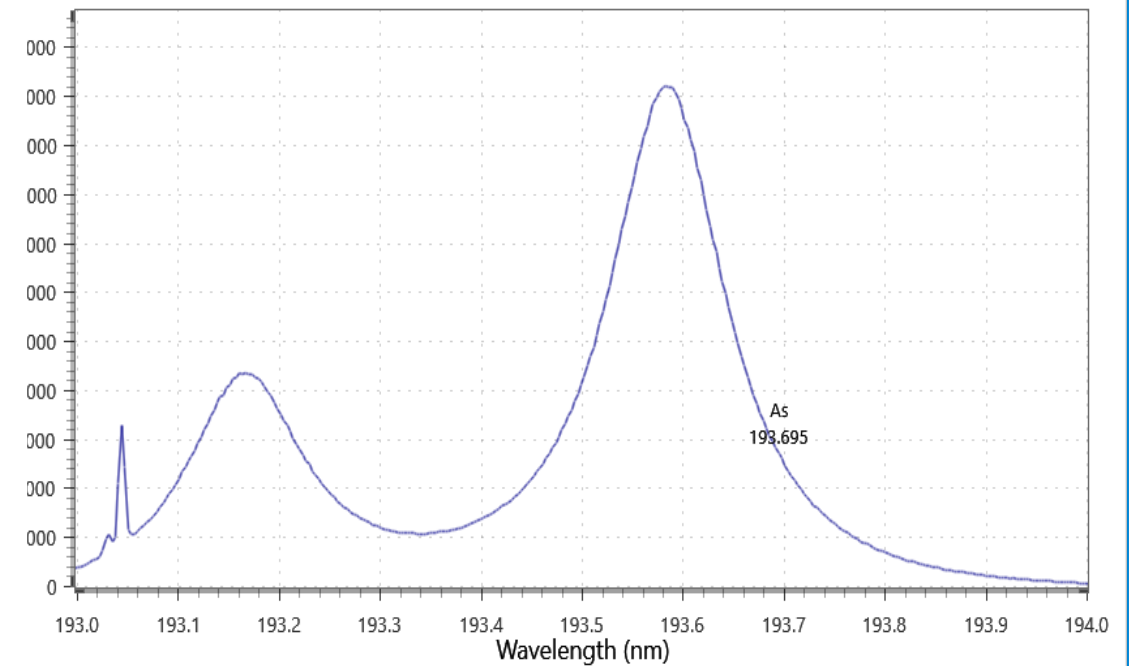
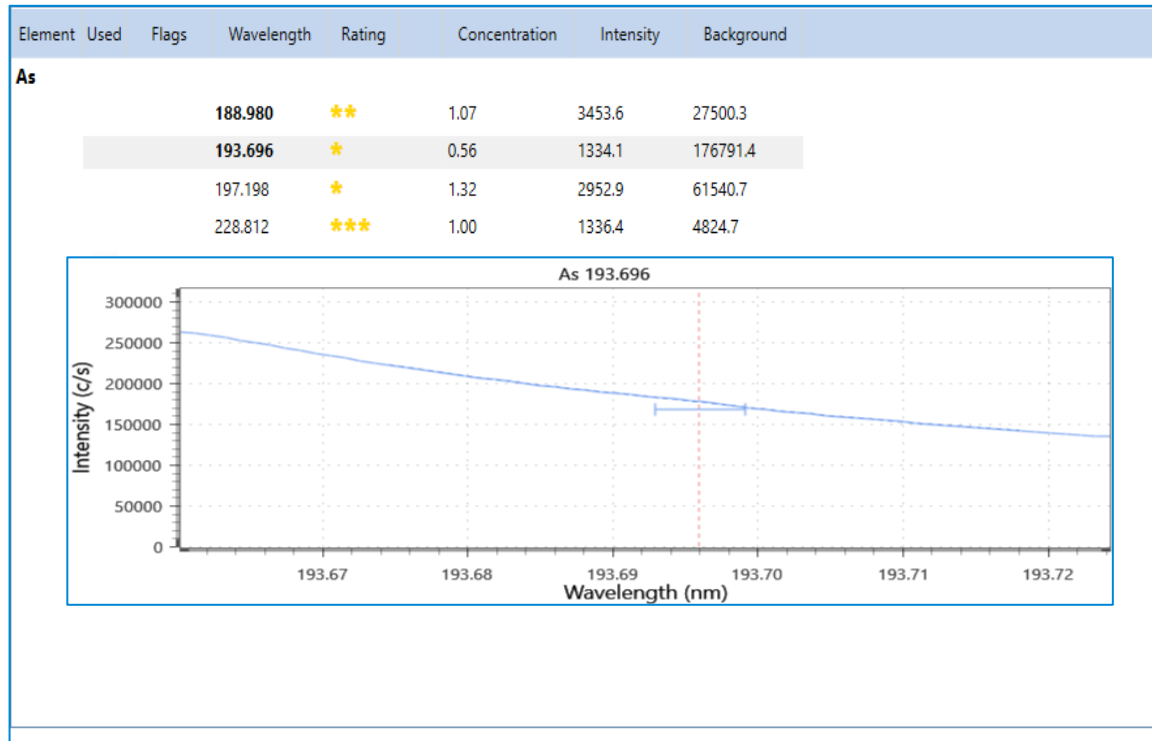
2 mg/L As in 5,000ppm Al (broad aluminum auto-ionizing doublet is clearly visible)

INSET – As wavelength scan at 193.696 nm

# Real World Examples

## Al interference on As in earth sample

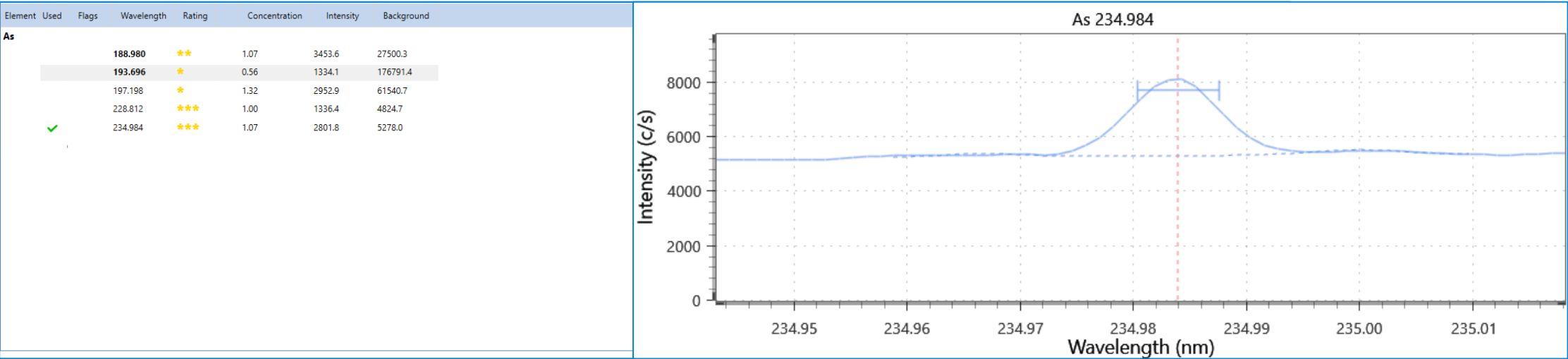
US EPA method 6010 recommends the As193.696 nm emission line



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 Al 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