Technical Overview

Agilent Trusted Answers

Agilent IntelliQuant for ICP-MS

Fast, automated semiquantitative ICP-MS analysis provides greater sample insight and confidence in the results

Smart tools simplify ICP-MS operation and data review

Analytical laboratories, particularly in the commercial sector, are under constant pressure to increase productivity and reduce costs, while ensuring that data quality is not compromised. Many such laboratories have adopted the fast, multi-element technique of ICP-MS to meet their inorganic (metals) analysis requirements. However, ICP-MS is still often thought of as a complicated technique, requiring an expert user to develop new methods and interpret the data. Agilent ICP-MS instruments are quick to learn and easy to use, with functions such as preset methods, autotuning, and a software interface that guides users through typical workflows. Developments including helium collision cell to control polyatomic interferences, Ultra High Matrix Introduction (UHMI) for increased matrix tolerance, and a wide dynamic range detector also contribute to a simpler overall analytical workflow. To provide users with the confidence that their methods are robust and their results are accurate, Agilent has developed a range of smart functions to simplify operation and data review. These features include IntelliQuant, a software function that provides deeper sample knowledge, informing decision making from sample preparation, method development, sample analysis, and data reporting.

What is IntelliQuant?

IntelliQuant is a fully integrated results display and interpretation function that provides extra insight into samples with minimal additional time or effort. IntelliQuant is based on full mass-spectrum data acquired using the Quick Scan feature in the ICP-MS MassHunter software and is included in version 4.6 and later of that software.

For ease-of use, IntelliQuant data is automatically acquired as part of most quantitative methods and is a default setting in all Agilent preset methods. IntelliQuant automatically acquires full mass Quick Scan data within 2 s and generates semiquantitative data for up to 78 elements in each sample. IntelliQuant data is acquired in helium cell mode by default, so analytes are largely free from polyatomic ion interferences, ensuring high quality data is generated. IntelliQuant can also process Quick Scan data collected using previous versions of ICP-MS MassHunter.

IntelliQuant data provides the analyst with:

- The approximate concentration of as many as 78 elements in each sample, including those elements not in the quantitative method, without the need to prepare or measure any additional standards.
- Identification or confirmation of elements not included in the quantitative method that may be of interest or concern from a quality control or safety perspective.
- An estimation of the total matrix solids (TMS) content of each sample, which can help to identify the cause of matrix effects that might affect sensitivity and recovery. TMS data can also flag samples that may need to be handled differently in subsequent analyses, for example when a batch of mixed waters unexpectedly includes some highly saline samples.
- Monitoring for the presence of elements, such as certain rare earth elements (REEs) that can cause doubly charged ion interferences on critical elements like arsenic (As) and selenium (Se) (*1–3*).
- Information about sample preparation errors that, if uncorrected, would affect the accuracy of the results.

How does IntelliQuant work?

Automatic calibration

IntelliQuant is easy to use and requires no setup or calibration by the operator. IntelliQuant automatically selects the appropriate calibration and ISTD solutions from the quantitative measurements to calculate accurate response factors for every element—no specific standards are needed. Relative response factors for the elements not included in the calibration are calculated based on the measured response for nearby elements. These accurate response factors, combined with helium cell mode to control polyatomic ions, ensure that IntelliQuant provides high quality data even for uncalibrated analytes.

IntelliQuant periodic table heat map display

The periodic table heat map provides a clear visual overview of the concentration of all elements in a sample, enabling rapid data assessment. Semiquantitative concentrations are calculated and displayed for all measurable elements, except for elements assigned as ISTDs.

Figure 1 shows the IntelliQuant periodic table heat map acquired for a powdered donut (*4*). The heat map display includes all elements present in the sample, not just those elements that were included in the calibration standards. The heat map shows a high concentration of Ti (labeled as food additive "TiO_2" on the packaging). In this case, Ti was not selected for quantitative measurement but its presence at high concentration is clearly apparent from the IntelliQuant heat map display.

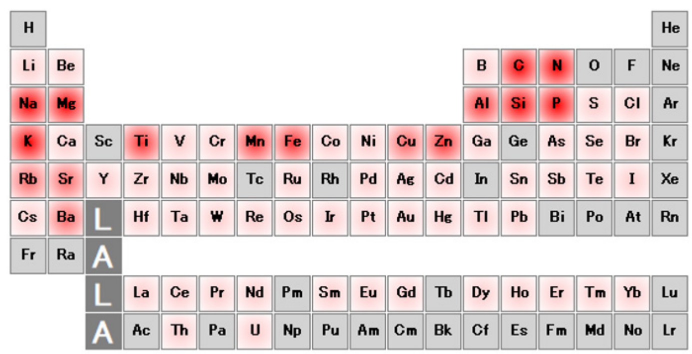

Figure 1. Periodic table heat map view of ICP-MS IntelliQuant data acquired for a powdered donut sample. The color intensity indicates the concentrations of elements within the sample i.e. the darker the red, the higher the concentration of that element.

IntelliQuant data can also help to identify sample preparation errors by identifying elements that should be present and are not, or vice versa. Sample preparation errors can lead to inadequate digestion or poor elemental stability for some elements, affecting the quality of the results. For example, HCl is routinely added to sample during samples preparation in order to stabilize analytes such as Hg, Ag, Sn, Sb, and Mo. The absence of Cl in the IntelliQuant heat map display gives a quick indication that the HCl addition might have been omitted from that sample.

Outlier results

The standard helium collision mode used on Agilent ICP-MS systems successfully resolves the polyatomic ion overlaps that can affect many analytes in typical samples. However, not all ICP-MS methods permit the use of He collision mode, and other non-polyatomic overlaps can also cause errors, especially in novel or unusual sample types. In these cases, IntelliQuant's outlier display helps the user to identify potential sources of error without needing a high level of ICP-MS expertise. The IntelliQuant outlier results can be displayed in the results table or in periodic table view, as shown in Figure 2.

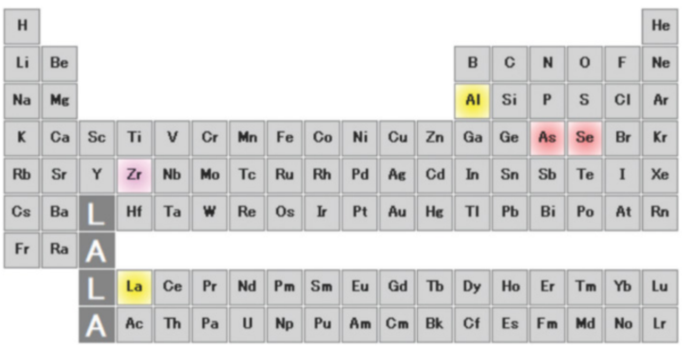

Figure 2. Color-coded outlier flags indicate analytes where intense signals at other masses could potentially cause spectral overlap.

Any analyte masses that are identified as being potentially impacted by other sample components are color-coded in the data table and highlighted in the periodic table, so they can be identified quickly. The outlier flags are based on the relative intensity of the analyte mass and the potential interference source, so they indicate potential overlaps rather than definite interferences. IntelliQuant scans the whole mass spectrum, checking for potential doubly-charged, adjacent mass, and polyatomic overlaps. By using the outliers in this way, users can be sure they are taking a cautious approach to ensuring the integrity of their data. The flags prompt the user to review and confirm any results that could be affected by unusual matrix components, so ensuring that false positive results do not slip through unchecked.

In the example shown in Figure 2, the yellow highlights for Al and La indicate the presence of an intense signal 16 u lower than the analyte mass, flagging the potential for a residual oxide overlap. Similarly, the pink shading on As, Se, and Zr flags the presence of an intense signal 40 u lower than the analyte mass, indicating the potential for residual argide overlaps. Other interference types such as dimers and doubly-charged ions are similarly color-coded. More information is provided by a "tool-tip" that is accessed by hovering the cursor over the element.

Determine the level of solids in a sample

The Total Matrix Solids (TMS) function uses the IntelliQuant data to calculate the approximate solids level of each sample. The calculation does not include gas or solvent elements, such as Ar, N, O, C, P, S, and halides, ensuring a more accurate result.

TMS is a powerful tool for method development for new or unusual sample types, enabling representative samples to be quickly assessed for total matrix level. TMS can also be used to track the sample load on the sample introduction consumables and interface cones, helping to keep on top of routine maintenance. Users of the 7850 ICP-MS can also use the early maintenance feedback (EMF) sensors and counters to determine when maintenance is needed, based on operation time or number of samples measured (*5*).

Agilent ICP-MS systems are routinely operated with very good plasma robustness (low CeO/Ce ratio), so are easily able to tolerate the level of dissolved solids present in most typical ICP-MS samples. However, very high matrix levels can cause signal changes by altering sample uptake and nebulization processes due to differences in sample viscosity. In the example shown in Figure 3, the ISTD signal was lower in samples 4 and 5, indicating a matrix effect in these samples. The ISTDs would correct for this change in sensitivity, so data accuracy would not be compromised. But the change in ISTD is a useful diagnostic tool, particularly when batches of unknown samples are analyzed. TMS data can provide more information on the samples that caused the lower ISTD signals. The TMS data for these two samples (Figure 4), showed that the matrix level of both samples was much higher than the other samples at around 1% (10,000 ppm). Using this information, the analyst might decide to flag these sample types to be diluted for future batches, or run with increased aerosol dilution using the UHMI system (*6, 7*).

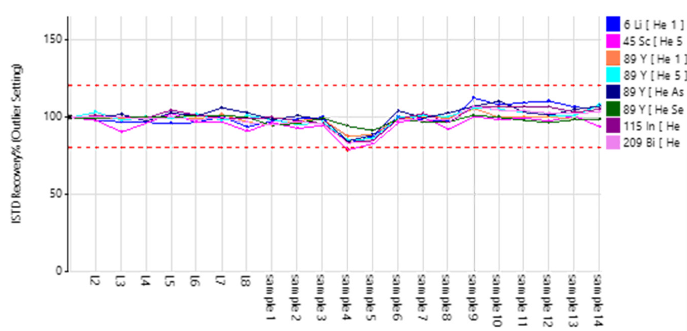

Figure 3. ISTD recovery plot for batch of variable matrix samples.

FullQuant	IntelliQuant					
	Sample					
	Rjct	Type	Level	Sample Name	TMS (ppm)	
9	☐	Sample		sample 1	1282.949	
10	☐	Sample		sample 2	1481.897	
11	☐	Sample		sample 3	2424.732	
12	☐	Sample		sample 4	13084.501	
13	☐	Sample		sample 5	9369.169	
14	☐	Sample		sample 6	2000.786	
15	☐	Sample		sample 7	1313.767	
16	☐	Sample		sample 8	3061.235	

Figure 4. TMS data for eight samples showing much higher matrix levels in samples 4 and 5.

Semiquantitative results

IntelliQuant provides semiquantitative data for up to 78 elements in a sample, including all elements in the full quantitative method as well as those elements not included in the calibration solutions. By providing semiquantitative data for every element, IntelliQuant gives extra confidence in the results. The data is especially useful when analyzing completely unknown samples, investigating contamination issues, or when troubleshooting data quality issues.

One example where IntelliQuant semiquantitative data can be useful is to prevent false-positive results being reported for important elements such as As and Se. If a sample contains REEs at a high enough concentration, doubly charged ion interferences can form that overlap the singly charged As and Se ions at half the mass of the REE. For example, $^{150}Nd^{2+}$ and $^{150}Sm^{2+}$ interfere with $^{75}As^{+}$, and $^{156}Gd^{2+}$ and $^{156}Dy^{2+}$ interfere with $^{78}Se^{+}$. In Elemental Analysis Method (EAM) 4.7 for ICP-MS, the US Food and Drug Administration (FDA) recommends that analysts monitor REEs to prevent bias of the As and Se results and use appropriate interference correction equations if needed (*3*).

To simplify the correction process for analysts, ICP-MS MassHunter software includes an easy-to-use "M^{2+} Correction" routine that automatically sets the parameters needed for the correction of REE doubly-charged ion interferences (*1*, *2*).

Isotope fingerprints

Since IntelliQuant obtains He mode data across the full mass spectrum from 2 to 260 u, data is collected for all isotopes of each element. If a sample result is flagged as unusual or unexpected, the Quick Scan data can be checked against the natural isotope abundance templates to confirm the presence of the queried element(s). To test this feature, a tap water sample was measured using IntelliQuant, and the peaks were checked for isotope abundance template fit, as shown in Figure 5. The spectrum of the tap water sample, which contained 200 ppm Na, shows a good fit of the natural isotope templates of all measured elements, including for ^{63}Cu and ^{65}Cu. The results confirm the effectiveness of He mode on the 7850 to remove the $^{23}Na^{40}Ar$ interference on ^{63}Cu.

The good isotopic template fit of the mass spectrum measured in He mode provides increased confidence in the analytical results without the need to remeasure the sample. IntelliQuant provides useful analytical insight into the sample that helps to validate the quantitative results.

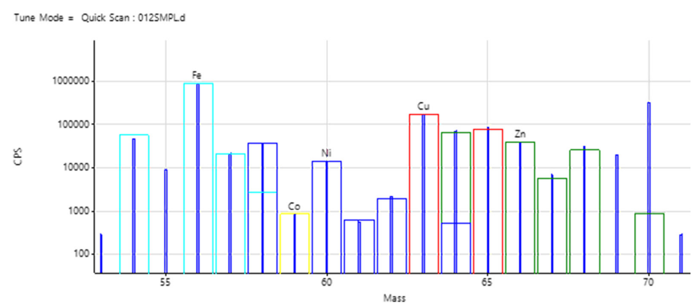

Figure 5. An IntelliQuant Quick Scan full mass spectrum enables confirmation of analyte/matrix element identity from isotope template match. In this example, the isotope template for Cu shows the removal of the $^{23}Na^{40}Ar$ interference on ^{63}Cu in helium cell mode.

Example of an IntelliQuant analysis

NIST 1643e Trace Elements in Water standard reference material (SRM) was analyzed using an Agilent 7850 ICP-MS. A preset method for low matrix samples and lens-autotuning were used to simplify the method development. When a preset method is used in ICP-MS MassHunter software, a Quick Scan acquisition is automatically collected in helium cell mode together with the quantitative measurements.

The quantitative analysis calibration standards were prepared using an Agilent mixed standard solution (p/n; 5183-4688). The ISTD solution was prepared at 1 ppm by diluting Agilent ISTD solution (p/n; 5188-6525) and single element standards for Ir and Y (Kanto Chemicals, Japan).

The accuracy of semiquantitative results is highest for analytes that are included in the standard used to calculate the response factors. But, even for elements that are not present in the standard, the interpolated response factors calculated automatically by IntelliQuant give a good indication of the true concentration. This is illustrated in Table 1, which shows the IntelliQuant results for NIST 1643e, calculated from the 2s Quick Scan data collected automatically as part of the acquisition.

The semiquantitative concentration results for all elements in the water SRM were within ±40% of the reference values, including for non-calibrated elements, as shown in Table 1.

Table 1. IntelliQuant semiquantitative recoveries for elements in 1643e water SRM.

Element	Measured Concentration (ppb)	Certified Concentration (ppb)	Recovery (%)
9 Be	13.7	13.98 ± 0.17	98
11 B	121	157.9 ± 3.9	77
23 Na	18100	20740 ± 260	87
24 Mg	7050	8037 ± 98	88
27 Al	122	141.8 ± 8.6	86
39 K	1350	2034 ± 29	66
43 Ca	22300	32300 ± 1100	69
51 V	35.6	37.86 ± 0.59	94
52 Cr	21.1	20.4 ± 0.24	103
55 Mn	38.8	38.97 ± 0.45	100
56 Fe	126	98.1 ± 1.4	129
59 Co	27.8	27.06 ± 0.32	103
60 Ni	56.9	62.41 ± 0.69	91
63 Cu	23.3	22.76 ± 0.31	102
66 Zn	80.6	78.5 ± 2.2	103
75 As	57.4	60.45 ± 0.72	95
78 Se	14.1	11.97 ± 0.14	118
85 Rb	9.19	14.14 ± 0.18	65
88 Sr	199	323.1 ± 3.6	61
95 Mo	120	121.4 ± 1.3	99
107 Ag	0.985	1.062 ± 0.075	93
111 Cd	5.77	6.568 ± 0.073	88
121 Sb	55.3	58.3 ± 0.61	95
137 Ba	454	544.2 ± 5.8	83
185 Re	106	113	94
205 Tl	7.11	7.445 ± 0.096	95
208 Pb	18.3	19.63 ± 0.21	93
232 Th	9.4	10*	94
238 U	10.0	10*	100

*Information value for Re. *Not included in 1643e SRM, so spiked at 10 ppb.*

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