Technical Overview

Agilent
Trusted Answers

Capabilities and Operation of the Advanced Dilution System 2

Automate calibration and sample dilution for Agilent ICP-OES and ICP-MS instruments

Improve the efficiency of your workflow

Now, more than ever, laboratories worldwide are facing increasing challenges to perform more analyses with fewer resources, prompting a re-evaluation of many analytical practices. Automation of certain tasks can help lab managers to streamline workflows and optimize resource utilization. To increase the efficiency of labs equipped with Agilent ICP-OES or ICP-MS instrumentation, Agilent has developed the Advanced Dilution System 2 (ADS 2)—a fully integrated inline autodilution system designed for routine, high throughput applications. The ADS 2 can automatically dilute stock standards and samples up to 400 times, making it ideal for the auto-preparation of calibration standards and samples, or dilution of overrange samples. The ADS 2 effectively reduces the number of manual tasks that operators have to perform when preparing a quantitative method for ICP-OES or ICP-MS, allowing more time to work on other tasks.

The two-syringe ADS 2 is available for the Agilent 5800 and 5900 ICP-OES* and Agilent 7850, 7900, and 8900 ICP-MS*. The ADS 2 and Advanced Valve System (AVS)** work seamlessly together under software control to maximize sample throughput, increase sample turn-around-time, and reduce cost-per-sample. The integrated design of the ADS 2 and AVS avoids adding excess time when not performing a dilution, addressing a common drawback of other dilution systems. The design also ensures that the system remains readily available to provide inline sample dilution whenever needed. If a reactive dilution of a sample is required, the intelligent software conveniently presents the results for the diluted sample at the end of each worksheet run, while retaining full access to all data.

Features of the ADS 2 autodilutor

The ADS 2 is fully integrated and controlled using Agilent ICP Expert software, version 7.7 and above, for ICP-OES and Agilent ICP-MS MassHunter software, version 5.3 and above, for ICP-MS.

The simple two-syringe autodilution system enables:

Autocalibration—the ADS 2 will automatically prepare calibration standards from accurate dilutions of a stock standard. The analyst simply places the stock standard onto the autosampler rack and uses the autocalibration assistant to define the calibration range. Multipoint calibration curves will then be automatically generated. The autocalibration assistant supports multiple stock standards and calibration ranges per element. Compared to manual processes, autocalibration is a convenient and efficient process that saves analyst time, minimizes waste, and reduces the risk of introducing errors or contamination into the analysis.

Prescriptive dilution—during method setup, the operator can set a defined (prescriptive) dilution factor for the automatic preparation of sample solutions. For example, if a series of sample solutions need to be diluted 10 times (1 in 10) before analysis, the analyst simply enters 10 as the dilution factor in the sample list. The software will then automatically trigger the ADS 2 to prepare the solutions prior to sample analysis.

Reactive dilution—following an unexpected result e.g., if the measured result for a sample is above the calibration range or if there is an internal standard recovery issue, the ADS 2 can be triggered to automatically perform a dilution. Based on the failed result, the software uses an algorithm to calculate an appropriate reactive dilution factor and triggers a rerun of the sample measurement. This automatic process eliminates the need for time-consuming, manual rework, by ensuring a complete data set at the end of a run. Samples measured at multiple dilutions are summarized according to the correct, in range result for each element. This software-assisted data review process enables quicker export of the results, simplifying the process for the analyst.

Faster turnaround times—when not actively diluting a solution, the ADS 2 has been optimized to ensure that almost no extra time is added to the analysis, typically less than two seconds. The flow of solution is directed through the dilution flow path of the ADS 2 only when a dilution is triggered (either defined in the sample list or reactively). The ADS 2 therefore provides the benefits of intelligent autodilution while maintaining fast sample turn-around-times similar to that of an ICP equipped with an AVS switching valve.

Reduced cost-per-analysis—with a simple two-syringe design and by only diluting samples and solutions when needed, the ADS 2 saves on consumables, like replacement syringes and valve wear parts. Consumption of labware, including gloves, sample vials, and pipette tips, and waste disposal costs, is reduced in comparison with manual dilutions. The cost-effective operation of the ADS 2 is further enhanced by its intelligent, software-controlled functionality saving analysis time and ICP operating costs (argon and power consumption etc.).

Ease-of-use—there are several tools in the ICP Expert and ICP-MS MassHunter instrument control software that streamline method development, data analysis, reporting, and troubleshooting when using the ADS 2. Both software suites include interactive flow path diagrams that provide real-time system information about the ADS 2 and the Help and Learning Center includes detailed information on how to use and easily maintain the system. Features including smart Early Maintenance Feedback (EMF) instrument performance-tracking counters and sensors, maintenance log, and online guides assist decision making, so that preventative maintenance is performed at the right time.

All from Agilent—The ADS 2 is optimized for Agilent ICPs, designed to work as an integrated system. All settings are included in the method, so there's only one software application to learn. Tight integration of the autodilutor with the instrument allows advanced features that can only be achieved when software and hardware are designed as one. The purchasing and support processes are simple as there's only one company to deal with.

**The ADS 2 is also compatible with the Agilent 5100 and 5110 ICP-OES and Agilent 7800 ICP-MS. **AVS for ICP-MS (AVS MS) formerly called ISIS 3.*

How does the ADS 2 Work?

The two syringes of the ADS 2 only actuate when needed to perform a dilution, maximizing the efficiency of the ICP workflow, to improve analysis turnaround times and cost-per-sample. As shown in Figures 1 and 2, the ADS 2 uses two distinct modes of operation (non-dilution and dilution).

In non-dilution mode (Figures 1a and b), the sample bypasses the ADS 2, maintaining the throughput of a switching valve method and maximizing the lifetime of the system components. In dilution mode (Figures 2a to d), a switch of the valves A and B brings the benefit of inline automatic dilution to the ICP analysis, removing manual processes including calibration preparation and sample dilution. The ADS 2 automatically injects a bubble between sample and carrier streams that prevents mixing with the carrier solution. This action maximizes the usable read time of the sample, minimizing the wash in/wash out time.

Valve A of the ADS 2 is used to direct flow between the AVS and valve C. Valve B is where the solution flows in from the autosampler and is directed directly to valve A or into the dilution loop. The syringes that contain the diluent and carrier solutions, never the sample solution, are connected to valve B. Valve C facilitates the rinsing of the ADS 2 system. Indicator lights positioned next to the valves A and B signify which function is taking place, whether loading (yellow) or injecting (green).

The ADS 2 and AVS also provide the flexibility to include or exclude the automatic online addition of internal standard during the analysis without the need for another syringe and associated costs.

Non-dilution mode

As shown in Figure 1a, the autosampler probe moves to uptake the sample (indicated in dark blue), where it is drawn by the AVS pump to load into the AVS sample loop, bypassing the dilution loop on the ADS 2. The AVS is now in the 'load' position. Any excess sample is delivered to waste by the AVS pump. At the same time, the peristaltic pump delivers the rinse solution (light blue) and internal standard solution (purple) to the nebulizer, spray chamber, and torch of the ICP instrument in preparation for delivery of the sample by the AVS. The two syringes of the ADS 2 are idle in non-dilution mode.

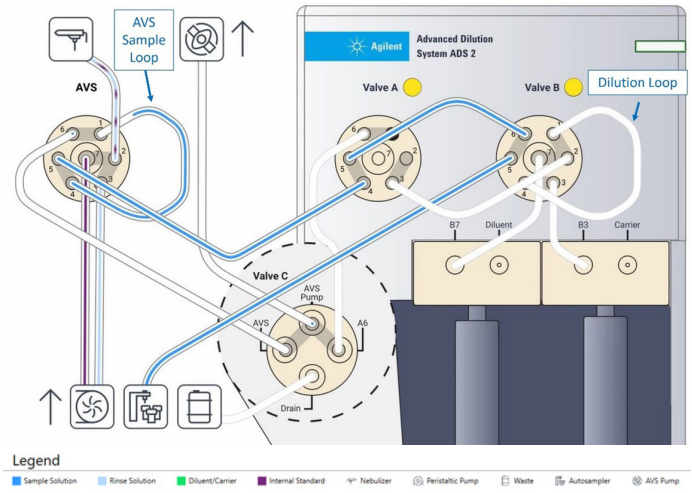

Figure 1a. Non-dilution mode: the sample is loaded into the AVS from the autosampler, bypassing the dilution loop of the ADS 2.

As shown in Figure 1b, when the AVS switches to the 'inject' position, the sample (dark blue) mixes with the internal standard (purple) and is pushed by the carrier solution (light blue) into the sample introduction system of the ICP. This process is conducted by the peristaltic pump. At the same time, the flow path to the autosampler is rinsed using the AVS pump through valve C (also light blue) in preparation for the next sample. The syringes of the ADS 2 remain in the idle position.

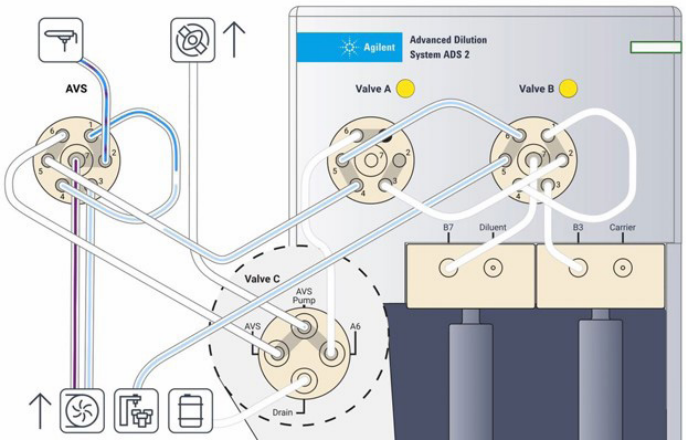

Figure 1b. Sample injection from the AVS to the Agilent ICP-OES or ICP-MS (no dilution).

Dilution mode

As shown in Figure 2a, the autosampler probe moves to uptake the sample (dark blue), where it is drawn by the AVS pump to load into the dilution loop of valve B of the ADS 2. Any excess sample is bypassed through valve C and delivered out to waste by the AVS pump. At the same time, the nebulizer, spray chamber, and torch are presented with the rinse (light blue) and internal standard (purple) solutions in preparation for delivery of the sample by the peristaltic pump. The AVS is now in the 'load' position.

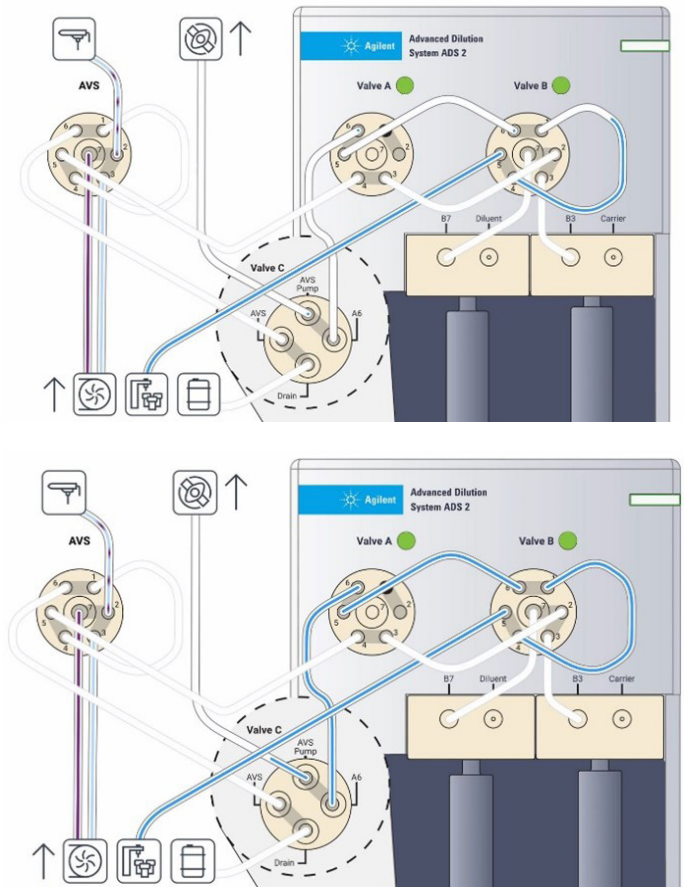

Figure 2a. Dilution mode: the sample starts to be loaded into the dilution loop of the ADS 2 (top) and once filled, any overflow is pumped to waste (bottom).

As shown in Figure 2b, valve B switches to the inject position and the diluent and carrier syringes begin to deliver their respective solutions into the valve/loop. The diluent (green) enters valve B at port 7, where it mixes at port 2, with the preloaded sample (dark blue) entering from the the dilution loop at port 1.

A small air bubble is injected to prevent the sample and carrier from mixing. This separation maximizes the solution available for measurement without the use of additional accessories. The carrier syringe delivers the carrier solution (also green) to push the sample through the dilution loop, without the carrier ever touching the sample solution. Throughout this process, the nebulizer, spray chamber, and torch are still being presented with the rinse (light blue) and internal standard (purple) solutions by the peristaltic pump in preparation for sample delivery. At this point, the AVS is still in the 'load' position.

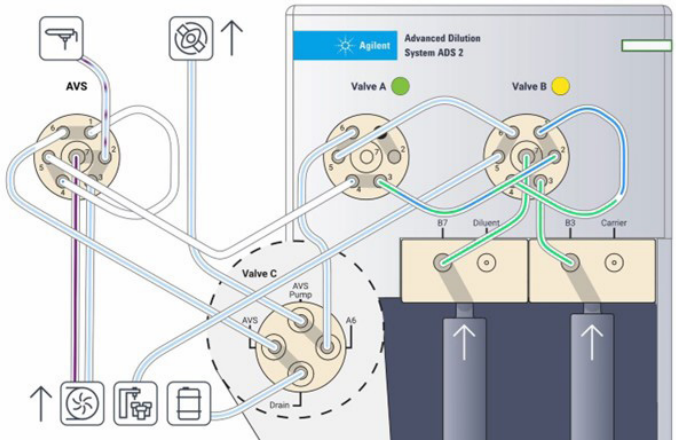

Figure 2b. Dilution process and loading of the AVS loop.

As shown in Figure 2c, the diluted sample (mixed dark blue/green) is directly transferred from valve B to valve A and loaded into the sample loop of the AVS. Any excess solution exits the AVS sample loop and goes to waste. The AVS valve is in the 'load' position.

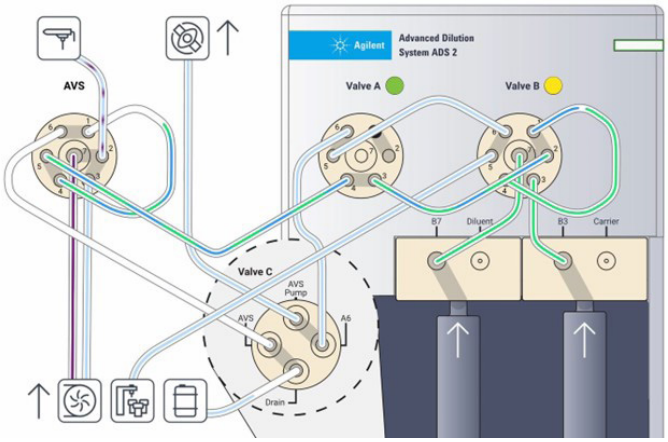

Figure 2c. Loading of the AVS loop with diluted sample.

As shown in Figure 2d, the AVS valve switches to the 'inject' position. The diluted sample (mixed dark blue and green) is mixed with the internal standard (if using) and is then delivered to the nebulizer using the peristaltic pump. Simultaneously the dilution loop and autosampler tubing is all rinsed, ready for the next sample.

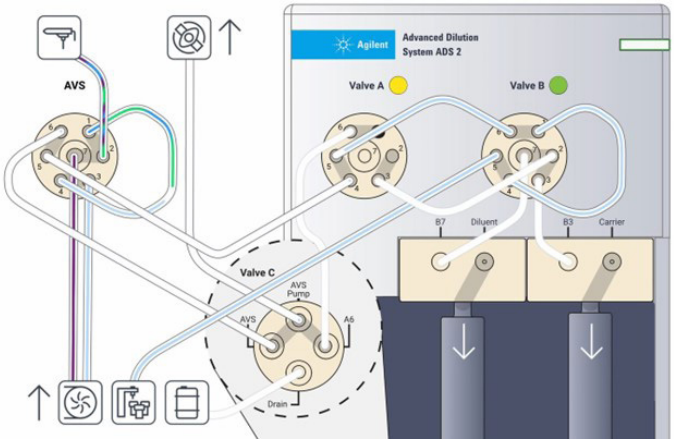

Figure 2d. Delivery of the diluted sample to the Agilent ICP-OES or ICP-MS.

Automatic calibration

The preparation of calibration standards is a critical step in collecting high-quality analytical data by ICP-OES or ICP-MS. To ensure data accuracy, calibration requires care and attention. In a poll conducted in 2023, preparing calibration standards ranked second in the manual tasks that consume most of an analyst's time.

Using the ADS 2 to automatically prepare calibration standards from a single multi-element stock standard or multiple standards accelerates the calibration process. Autocalibration also minimizes the risks of errors and contamination associated with manual preparation methods.

Both the ICP Expert and ICP-MS MassHunter software packages include an 'Autocalibration Assistant', example shown in Figure 3. The 'stock library' in the software includes a list of common calibration stock standards. Custom standards can be easily added to the library. By simply selecting a stock standard from the library, and inputting a dilution factor, the calibration concentrations are calculated automatically, and the ADS 2 takes care of calibrating the ICP.

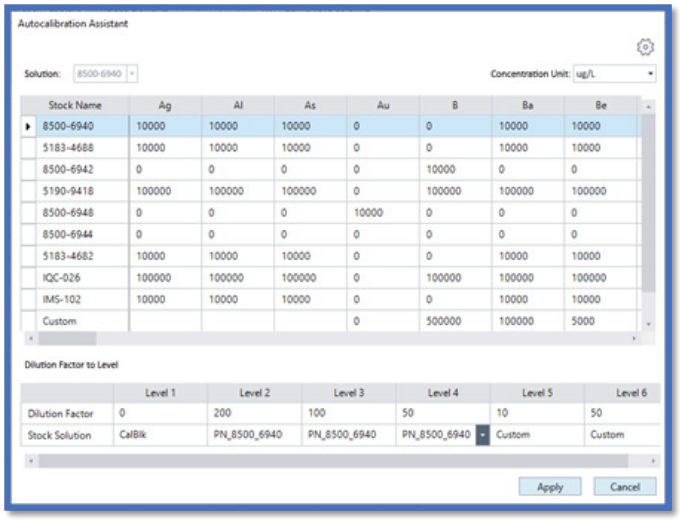

Figure 3. Stock standards library (top), and the automatic calculation of calibration levels from the dilution factor prescribed to the stock solution (bottom).

Automating the preparation of calibration standards also removes any operator-to-operator variability that is inherent in manual processes, enhancing ICP data quality in the lab. The automatically prepared calibration standards generate linear calibration curves across a wide analytical range, with correlation coefficients (R) typically above 0.9999 and with <5% error on each point.

A representative ICP-MS calibration curve for thallium (^{205}Tl) from 0.25 to 100 µg/L is shown in Figure 4. The excellent linearity of the lower-level calibration standards (zoomed scale, right) shows that the ADS 2 can accurately dilute standards up to 400x.

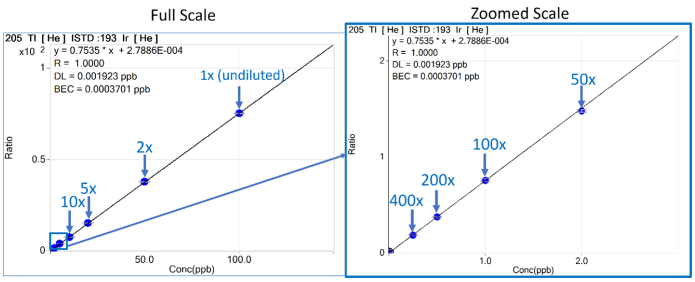

Figure 4. Left: ICP-MS calibration curve for ^{205}Tl from 0.25 to 100 µg/L with excellent correlation coefficient of R= 1.0000 generated in Agilent ICP-MS MassHunter software. Right: A zoomed in section of the lower concentration calibration standards prepared using the ADS 2 from 400x to 50x.

A representative ICP-OES calibration curve for Se 196.026 nm from 0.0125 to 5 mg/L is shown in Figure 5. The data shows an excellent correlation coefficient of R = 1.0000 was obtained with <4% error, further demonstrating the capabilities of the ADS 2 for the accurate dilution of standards up to 400x.

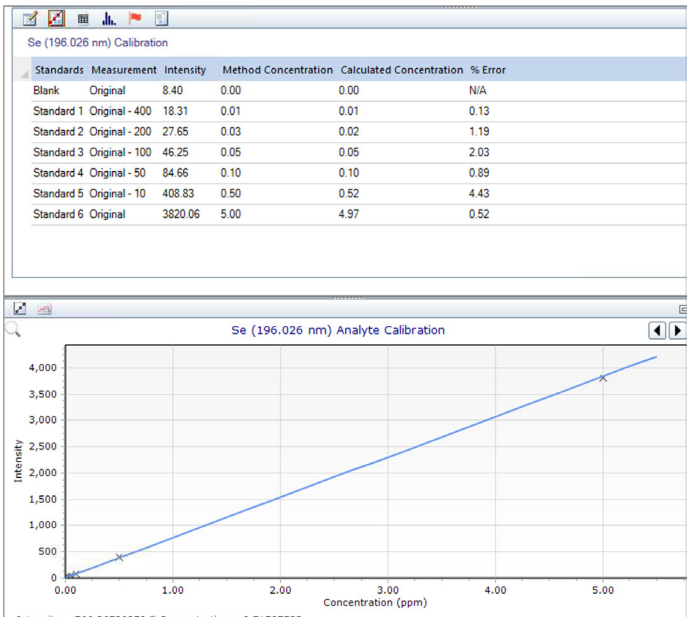

Figure 5. ICP-OES calibration curve Se 196.026 nm from 0.0125 to 5 mg/L with excellent correlation coefficient of R = 1.0000 generated in Agilent ICP Expert software.

Autocalibration by the ADS 2 ensures that fresh standards are used for each analysis, improving data quality, while also reducing the amount of waste produced from manual standard preparation.

Prescriptive dilution

The ADS 2 can save analyst time by automating the tedious and repeatable manual task of diluting samples before analysis. Once predefined dilution factors from 2x to 400x have been selected in the instrument control software, the ADS 2 will automatically dilute the samples. Prescriptive dilution removes the need to manually dilute samples before measurement, freeing up analysts to work on more valuable tasks. The ADS 2 prepares samples with high repeatability, removing the risk of error associated with manual dilution procedures.

Prescriptive dilution can also be applied to QC solutions such as certified reference materials (CRMs). For example, the ADS 2 can apply the same dilution factor used for a sample to a CRM.

Reactive dilution

Sample remeasurement is one of the top five manual handling tasks that increases sample turnaround time and cost-per-analysis in a poll conducted in 2023.

ICP Expert or ICP-MS MassHunter instrument control software packages can automatically determine when a sample result is out of range. Unexpected results may include a result that is outside of the calibration range, or a result where the internal standard ratio is outside of the limits set by the analyst. In these cases, the software triggers the ADS 2 to automatically dilute the sample for remeasurement, requiring no user intervention. This approach simplifies the analysis, reduces the cost of manually diluting and remeasuring a sample, and ensures swift turnaround times, all while maintaining the accuracy of reportable results. Reactive dilution is also available if a QC solution fails.

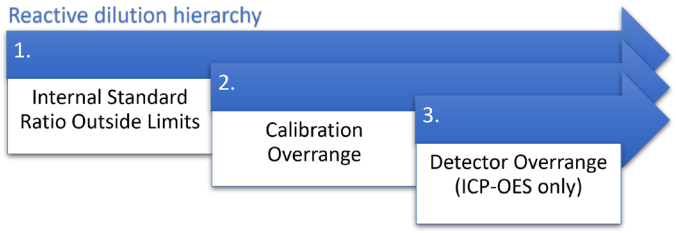

Figure 6. Reactive dilution decision process within Agilent ICP Expert and ICP-MS MassHunter software packages.

Simple and smart

Both ICP Expert and ICP-MS MassHunter software suites include smart Dilution Lists within the ADS 2 functionality. Dilution Lists can provide rules for action based on an overrange result for a sample, a failed QC, or if an internal standard ratio falls outside of the desired limits for a set of key analytes.

Controlling dilution triggers for different sample types

As shown in Figures 7 and 8, the Dilution List function provides the flexibility to only dilute samples based on overrange results of selected key elements. This list can then be applied to samples on an individual basis, preventing unnecessary measurements. The function guarantees fast turnaround times and reduces the cost-per-sample.

As an example, some labs want to measure a batch with different water types in a single analytical method, an analyst may not want to report a result for sodium (Na) in seawater but would want to include Na as an analyte during the analysis of drinking water samples. By setting up a Dilution List that excludes Na from being a dilution trigger for seawater samples, the software can ensure that no action is taken, avoiding unnecessary dilution and remeasurement. However, for drinking water the ADS 2 would automatically (reactively) dilute the sample.

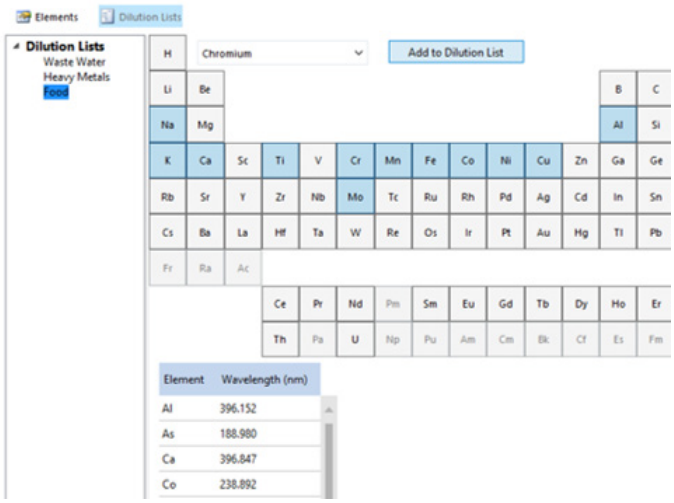

Figure 7. Dilution List configuration tab in Agilent ICP Expert 7.7 software.

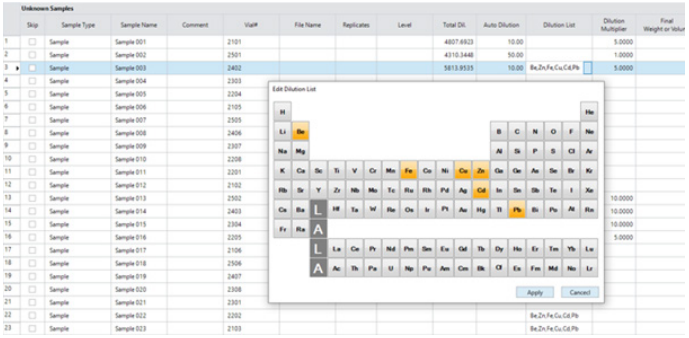

Figure 8. Dilution List element selection pop-up window in Agilent ICP-MS MassHunter 5.3 software.

Automatic collation of the best results for each sample

The Summary feature of the ADS 2 software for ICP Expert and ICP-MS MassHunter simplifies and automates data analysis and reporting. The result summary uses a smart algorithm to filter all measurements of a sample and presents the best result for each element, as outlined in Figure 9 for Mg and Fe. The single summarized result for each analyte in each sample can be easily exported from the instrument software into the report template. An example of a Summary view of ICP-OES sample data for Al, As, Ba, and Fe is shown in Figure 10. All data for all samples is retained and is available for export.

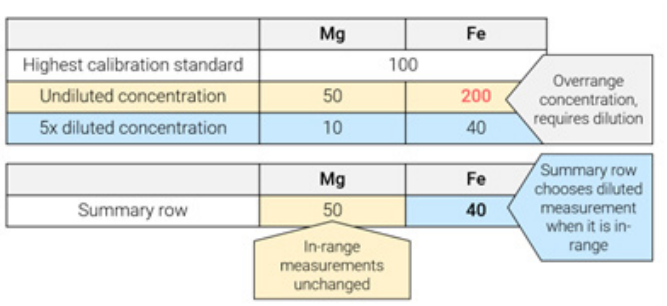

Figure 9. Summary feature decision tree for data reporting. (Note: All results presented using unadjusted values).

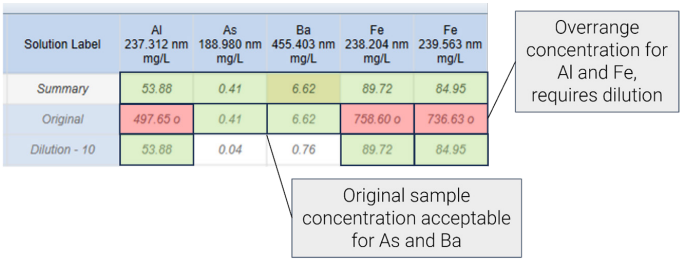

Figure 10. Example of sample data from Agilent ICP Expert showing the simplified Summary Row view of the best results for each analyte.

Variable sample volumes

The ADS 2 can be fitted with sample loops with volumes ranging from 0.5 to 3.0 mL, to suit the available sample volume. The choice of loop leads to measurement times of between 20 and 150 s by ICP-OES or 25 and 410 s by ICP-MS, as shown in Figure 11. The design and integrated control of the ADS 2 ensures consistent measurement times in both non-dilution and dilution modes of operation.

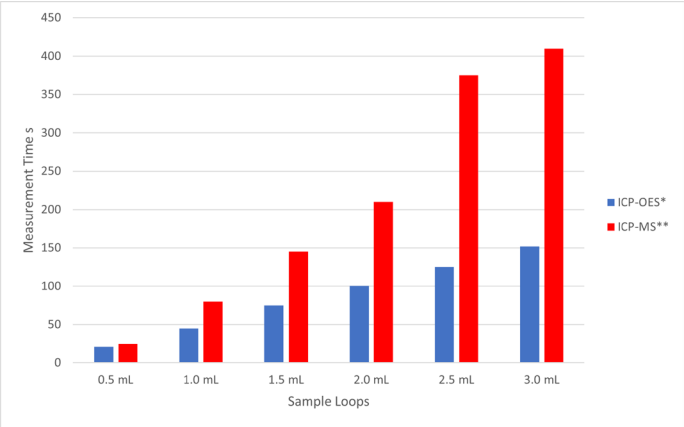

Figure 11. Maximum ICP-OES and ICP-MS measurement times using different-sized loops, and thus different sample volumes, for the ADS 2.

**ICP-OES measurement time based on 5 s stabilization time, 12 RPM peristaltic pump speed, and 1.02 mm white/white peristaltic pump tubing. ** ICP-MS measurement time based on 20 s stabilization time, 0.1 RPS peristaltic pump speed, and 1.02 mm ID white/white tubing.*

Useful method development tools

Both ICP Expert and ICP-MS MassHunter software packages include the following smart tools that are designed to facilitate method development:

– Conditions Calculator—a useful tool that provides recommended timings on method parameters from defined tubing type and length.

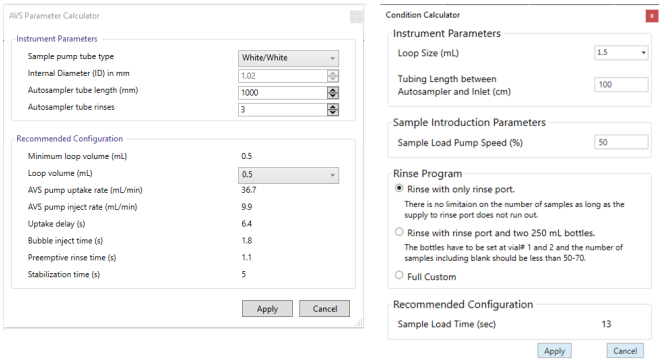

Figure 12. The Conditions Calculator in Agilent ICP Expert (left) and Agilent ICP-MS MassHunter software (right).

– AVS/ADS Timing Monitor—to check or further optimize method conditions, the AVS/ADS Timing Monitor function shows the acquired signal during the whole method sequence. For example, if the signal is stable sooner than set by the conditions calculator for a certain sample type, then the stabilization time could be shortened, saving time. The analyte signal is measured, and each condition change is flagged by the software, as shown in Figure 13 for the measurement of Zn 213.857 nm by ICP-OES. This tool is also useful for troubleshooting any potential issues within the system.

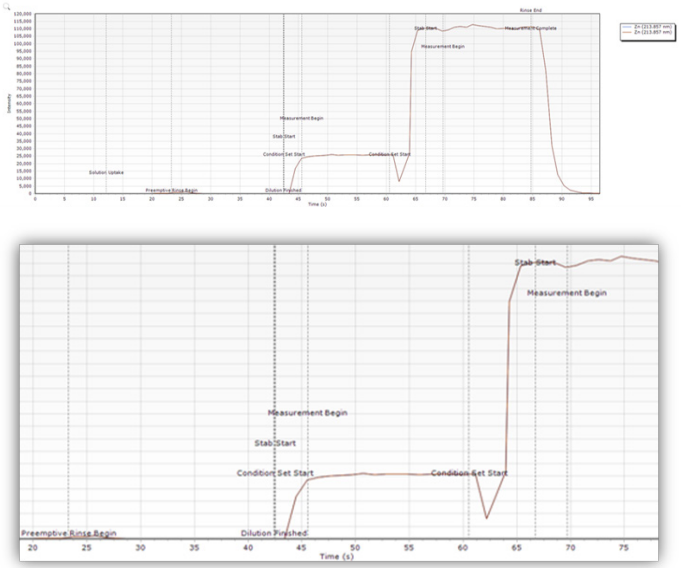

Figure 13. Agilent ICP Expert ADS/AVS Timing Monitoring example for the measurement of Zn 213.857 nm in a diluted sample for a two condition ICP-OES method, providing an overview of the conditions for the analysis. The two conditions relate to the radial measurement followed by the axial measurement of the signal. The >5 s stabilization time between setting axial conditions and beginning measurement could be reduced. Top: Full sequence. Bottom: Zoomed view between 20 and 75 s.

Reducing cost-per-sample and preventing contamination

Samples, such as some environmental waters collected in the field, can be sampled directly into 15 or 50 mL autosampler tubes and placed on an autosampler rack, ready to be automatically diluted. This approach eliminates the need for aliquot transfer and dilution in the lab, thereby avoiding unnecessary sample handling. Additionally, for samples needing multiple dilutions, either prescribed or reactive, only one vial is used. This efficient sample handling process contributes to the fast sample turnaround times, reduced risk of sample contamination and mistakes, and lower cost-per-sample associated with the ADS 2. A further advantage of streamlining time-consuming and repetitive manual tasks in the lab is the reduction of physical fatigue experienced by staff.

By removing manual dilution steps, the ADS 2 increases productivity, reduces energy consumption, and reduces waste of reagents and plastic consumables like pipette tips, sample vials, and gloves. All these factors combine to lower the cost-of-analysis and reduce the environmental impact of the analysis, helping labs to become more sustainable.

Figure 14. Removing manual sample dilution steps can help reduce plastic waste.

Troubleshooting and maintenance

The integration of the ADS 2 within the ICP Expert and ICP-MS MassHunter instrument software suites provides full control of the accessory, status monitoring, maintenance tracking, and enhanced troubleshooting capabilities.

An interactive flow path diagram that shows the movement of sample, rinse, diluent, carrier, and internal standard solutions through the autodilution system, in real time, is embedded in the ICP instrument control software (Figure 15). The flow path shows the solutions at each stage of the analysis. Therefore, if there is a blockage, the diagram can assist in identifying where the solution should be flowing, and where a potential blockage exists, simplifying troubleshooting.

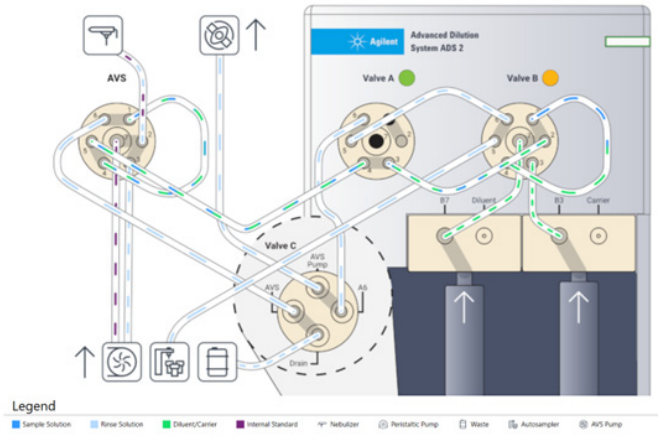

Figure 15. Interactive flow path diagram that shows the movement of the various solutions through the autodilution system to the instrument sample introduction system.

The AVS/ADS Timing Monitor can also be used to help troubleshoot issues. For example, if there is a leak in the tubing, or if the volume of diluent is low, or if the diluent bottle is empty, the timing monitor will visually indicate a problem. The trace of the signal can then be compared to a library of traces that represent common causes for signal-issues in the Help and Learning Center.

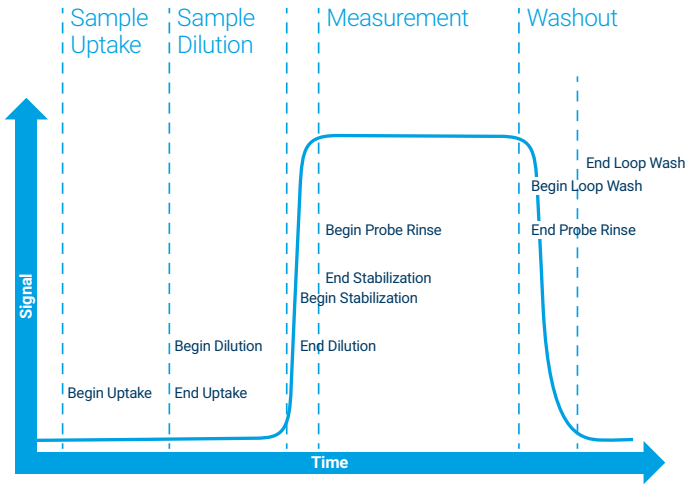

Figure 16. The software includes automated functions to determine the optimum method settings, based on the tubing lengths and sample loop size entered. The functions also monitor the measurement signal to provide information to fine-tune method settings or assist with troubleshooting.

Early Maintenance Feedback (EMF)

EMF tracks components of the ICP instrument, AVS, and ADS 2 and alerts operators when maintenance is required. Traffic light color-coding of the EMF counters show which maintenance activities should be done immediately (red), which are imminent (amber), and which can wait (green), as shown in Figure 17. The default settings for the counters are useful for most general applications, but users can set the counter limits to suit their specific requirements. EMF reduces downtime and repair costs by scheduling routine maintenance of components based on actual use, rather than at set time intervals.

As the ADS 2 only drives the syringes and switching valves when a dilution is being performed, EMF tracking ensures that maintenance is only performed when needed, rather than being based on elapsed time.

The maintenance log within the EMF function digitally records the maintenance history of the hardware, making it easy to determine if the instrument has been sufficiently maintained.

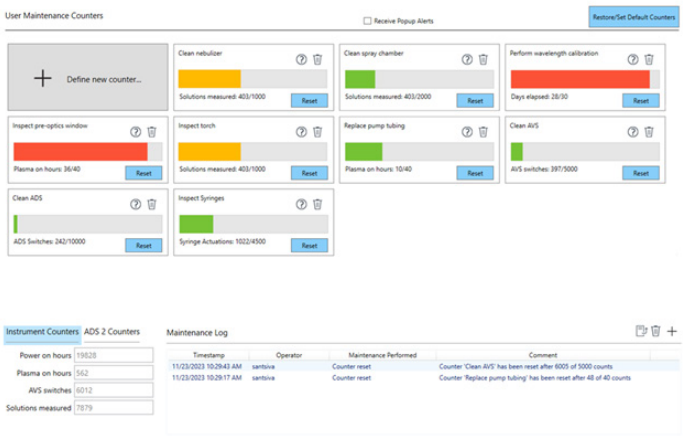

Figure 17. Example of an EMF screenshot showing maintenance counters for an Agilent ICP-OES, AVS, and ADS 2.

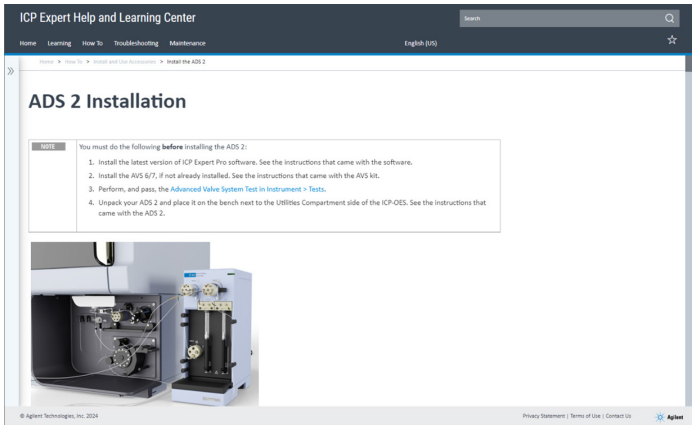

Figure 18. Access installation, operation, maintenance, and troubleshooting procedures for the ADS 2 in the integrated Help and Learning Center software pages of ICP Expert and ICP-MS MassHunter software suites.

Help and Learning center

To help analysts develop good practices when using the ADS 2, the Help and Learning center contains how-to guides and detailed videos on the operation, maintenance, and troubleshooting of the accessory. Both ICP Expert and ICP-MS MassHunter software suites include a quick access button to the Help and Learning center, which is positioned in the top-right corner of the instrument software screen.

Specifications

Dilution Range	2—400x
Syringe pump accuracy	± 1% @ 100% stroke
Syringe pump precision	≤ 0.05% @ 100% stroke
Dimensions	Height 37.9 cm (15 inches) Width 15.8 cm (6.2 inches) Depth 31.3 cm (12.3 inches)
Weight	7.9 kg (17.4 lbs)
Altitude	Up to 2,000 m
Compatibility	Agilent 5900, 5800, 5110 ICP-OES Agilent 8900, 7900, 7850, 7800 ICP-MS
Autosampler	Agilent SPS 4, SPS 6 or other autosampler supported in instrument software
Software	Requires MassHunter 5.3 or higher for ICP-MS Requires ICP Expert 7.7 or higher for ICP-OES

More information

1. McCarthy, D., Automating the Workflow for the Analysis of Soils by ICP-OES, Agilent publication, 5994-7203EN
2. Bradford, R., Determination of Multiple Elements in Lithium Salts using Autodilution with ICP-OES, Agilent publication, 5994-7179EN
3. Zou, A. Yamanaka, M., Intelligent Analysis of Wastewaters using an Agilent ICP-MS with Integrated Autodilutor, Agilent publication, 5994-7113EN
4. Yamashita, R., Automated Analysis of Low-to-High Matrix Environmental Samples Using a Single ICP-MS Method, Agilent publication, 5994-7114EN
5. Riles, P., Productive Analysis of High Matrix Samples using ICP-MS with Integrated Advanced Dilution System, Agilent publication, 5994-7232EN

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