Application Note
Environmental

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Fast and Robust Analysis of Various Types of Waters by ICP-OES Following Method HJ 776-2015

Using smart tools to facilitate the analytical performance of the Agilent 5800 VDV ICP-OES

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Introduction

Clean, safe water is fundamental to public and environmental health (*1*). The purity of groundwater and surface waters, such as lakes, river, and reservoirs, is important for drinking water supplies and for nature to thrive. However, these waters are often subject to pollution from industrial processes, agricultural practices, sewage overflow, or events such as wildfires or flooding, which impacts the quality of watercourses. Leading to an ongoing need to test various types of water for a range of contaminants, including elemental pollutants. Many countries and international bodies publish standard analytical methods to help companies and organizations meet their environmental regulatory requirements. For example, there are several standard methods that outline the analysis of environmental waters by ICP-OES, including the Chinese method HJ 776-2015 (*2*). HJ 776-2015 describes the analysis of 32 elements in surface water, groundwater, sewage, and industrial wastewater. To analyze a range of water samples, laboratories need methods that are suitable for low-level analysis of surface and groundwater, with the linear dynamic range (LDR) needed for wastewater analysis.

In this study, the Agilent 5800 Vertical Dual View (VDV) ICP-OES fitted with an Agilent Advanced Valve System (AVS) 7 port was used to analyze various water samples according to HJ 776-2015. The elements analyzed in this study include silver, aluminum, arsenic, boron, barium, beryllium, bismuth, calcium, cadmium, cobalt, chromium, copper, iron, potassium, lithium, magnesium, manganese, molybdenum, sodium, nickel, phosphorus, lead, sulfur, antimony, selenium, silicon, tin, strontium, titanium, vanadium, zinc, and zirconium.

Experimental

Instrumentation

The Agilent 5800 VDV ICP-OES was equipped with a SeaSpray concentric nebulizer, double-pass glass cyclonic spray chamber, and an Easy-fit demountable VDV torch with a 1.8 mm injector. For fast sample analysis and high sample throughput, an Agilent AVS 7-port switching valve and an Agilent SPS 4 Autosampler were used to deliver the samples to the instrument. The AVS 7, which is fully integrated into the sample introduction system of the ICP-OES, uses a high-speed positive displacement pump to rapidly fill the sample loop, speeding up the analysis. The AVS 7 also reduces the sample matrix reaching the sample introduction system, so less maintenance and cleaning tasks are needed when analyzing high matrix samples compared to conventional sample introduction. A range of smart tools within the Agilent ICP Expert Software help laboratories to run their samples more efficiently (*3*). From performing daily checks, preparing calibration standards and samples, developing methods, optimizing and verifying results, to data reporting; the tools streamline the analytical workflow.

- IntelliQuant provides a semiquantitative data set for every sample within seconds, which allows analysts to quickly verify the quantitative results and to identify elements not included in the quantitative method (*4*).
- Neb Alert immediately alerts analysts if there is a nebulizer leakage or blockage using sensors that monitor nebulizer argon pressure (*5*).
- Early maintenance feedback allows the user to set up alerts to prompt maintenance after a specified number of samples has been run (*6*). Tracking instrument health and carrying out maintenance tasks at the right time ensures consistent, high-level analytical performance, even of complex samples such as wastewaters.

Environmental waters often contain elements at various concentrations, ranging from low parts per billion (ppb) for surface waters to percentage levels in industrial wastewaters. Therefore, the ICP-OES needs a large dynamic range to avoid excess sample dilution steps during the analysis of unknown water samples. To ensure the wide LDR required for the application, the 5800 and 5900 ICP-OES feature the Agilent Vista Chip III charge coupled device (CCD) detector. The detector provides full wavelength coverage, enabling measurements of many different wavelengths per element to cover all concentration ranges in different water samples. The wide dynamic range reduces the need to dilute and remeasure overrange samples, saving a lot of analyst-time.

Also, since different wavelengths often have different sensitivities, a combination of wavelengths can be used for the same element, further extending the dynamic range of the method. Selecting the most sensitive line will give the best detection limit and accurate measurement of low-level analytes, while less sensitive lines allow the measurement of higher concentration analytes in the same measurement.

The MultiCal function within the ICP Expert Software creates multiple calibration ranges for an element, extending the dynamic range of the determination. Figure 1 shows how two wavelengths were used for Zn, providing a linear calibration up to 10,000 ppm. The wavelengths included Zn 213.857 nm (axial) for low concentration results and Zn 334.502 nm (axial) for high concentration results.

Figure 1. In this example, MultiCal uses Zn 213.857 nm (axial) to measure concentrations up to 60 mg/L (top), and the 334.502 nm (axial) line for higher concentrations, linear up to 10,000 mg/L (bottom).

An internal standard (5 mg/L of yttrium) was used to correct for matrix effects. The 5800 VDV ICP-OES operating conditions and AVS 7 parameters are listed in Tables 1 and 2.

Table 1. Agilent 5800 VDV ICP-OES operating conditions.

Parameter	Axial	Radial
Read Time (s)	10	2
Replicates	3	
Stabilization Time (s)	8	0
Pump Speed (rpm)	12	
RF Power (kW)	1.5	
Aux Flow (L/min)	1	
Plasma Flow (L/min)	12	
Nebulizer Flow (L/min)	0.7	
Viewing Height (mm)	NA	10
Sample Pump Tubing	White-white	
Internal Standard Pump Tubing	Black-black	
Waste Pump Tubing	Blue-blue	
Background Correction	Fitted	

Table 2. AVS 7 switching valve parameters.

Parameter	Setting
Sample Loop Size (mL)	1
Pump Rate - Uptake (ml/min)	36.0
Pump Rate - Inject (mL/min)	5.0
Valve Uptake Delay (s)	4.5
Bubble Injection Time (s)	2.0
Pre-emptive Rinse Time (s)	1.0

Reference materials and sample

Two Certified Reference Materials (CRMs) were used to validate the method: Certified Waste Water – Trace Metals Solution D (CWW-TM-D) (High Purity Standards, Charleston, South Carolina, USA) and NCS ZC 76307 (China National Analysis of Iron and Steel, Beijing, China). A household wastewater sample was also analyzed as part of the method development.

Sample preparation

The CRMs and household wastewater sample were prepared in accordance with the guidelines described in HJ 776 (*2*). HJ 776 advises that samples are acidified in a 1% nitric acid solution before preparation using the acid microwave digestion procedure outlined in HJ 678-2013. 25 mL of sample was added to a digestion vessel followed by 1 mL of 30% hydrogen peroxide (Emsure, Merck) and 5 mL of nitric acid (Emsure, Merck). The solution was then digested according to the temperature program detailed in Table 3 using a CEM MARS 6 Microwave Digestion System (CEM Corporation, NC, USA). The digests were diluted to a volume of 50 mL using 18.5 MΩ de-ionized (DI) water (Milli-Q IQ 7010 Water Purification System), resulting in a final matrix of 10% nitric acid and a dilution factor of 2.02.

Table 3. Temperature program for acid microwave digestion.

Parameter	Setting
Power (W)	1200
Temperature (°C)	180
Ramp Time (min)	10
Hold Time (min)	15

Calibration

The concentrations of the working calibration standards are shown in Table 4. All standards were prepared from Agilent single element stock solutions in a matrix of 10% nitric acid. The elements were grouped according to their chemical compatibility. Since the highest standard for all elements was either 250 or 500 mg/L, careful consideration was required to avoid the formation of precipitates. For example, the addition of As and Zr at high concentrations will form a precipitate. All wavelengths used either a one-point or two-point calibration. The lowest calibration point was required for trace level measurements. The highest concentration point, either 250 or 500 mg/L, reflects the guidelines provided in HJ 776.

Table 4. Concentration of working calibration standards.

Element	Std 1 (mg/L)	Std 2 (mg/L)	Std 3 (mg/L)	Std 4 (mg/L)	Std 5 (mg/L)	Std 6 (mg/L)	Std 7 (mg/L)	Std 8 (mg/L)
Ag	2					250		
Al	0.6		250					
As	1				500			
B	0.6			250				
Ba	0.6			250				
Be	0.15					250		
Bi	0.6					250		
Ca		0.6	250					
Cd	0.15			250				
Co	0.6					250		
Cr	0.6					250		
Cu	0.6					250		
Fe	0.6		250					
K		20	500					
Li		15	500					
Mg		0.6	500					
Mn	0.6					500		
Mo	0.6							500
Na		20	500					
Ni	0.6					500		
P	0.6		500					
Pb	0.6				500			
S	0.6					500		
Sb	1			500				
Se	1					500		
Si	0.6			250				
Sn				2			500	
Sr		0.6	500					
Ti	0.6					250		
V	0.6					500		
Zn	3				500			
Zr	0.6						500	

Linear Dynamic Range (LDR)

The LDR concertation given in Table 5 corresponds to the maximum concentration measured for each element/ wavelength. Linear concentrations for all elements met the specified concentrations listed in the HJ 776 method.

Table 5. The linear dynamic range concentration for all measured wavelengths.

Element, Wavelength (nm) and Viewing mode	LDR Concentration (mg/L)	Element, Wavelength (nm) and Viewing mode	LDR Concentration (mg/L)
Ag 328.068 Axial	250	Mn 259.372 Axial	60
Al 396.152 Axial	30	Mn 260.568 Radial	1000
Al 237.312 Axial	10000	Mo 202.032 Axial	250
As 188.980 Axial	500	Mo 277.539 Axial	1000
As 234.984 Axial	2500	Na 588.995 Axial	60
B 249.772 Axial	250	Na 588.995 Radial	1000
Ba 455.403 Axial	15	Ni 221.648 Axial	125
Ba 389.178 Axial	750	Ni 231.096 Axial	500
Be 234.861 Axial	30	P 214.914 Axial	750
Be 249.473 Axial	1000	Pb 220.353 Axial	500
Bi 223.061 Axial	1000	S 180.669 Axial	750
Ca 393.366 Axial	7	Sb 217.582 Axial	750
Ca 315.887 Axial	1000	Se 196.026 Axial	500
Cd 214.439 Axial	15	Si 251.611 Axial	250
Cd 226.502 Radial	1000	Si 185.005 Axial	500
Co 238.892 Axial	125	Sn 189.925 Axial	250
Co 230.786 Axial	500	Sn 181.059 Axial	750
Cr 205.560 Axial	60	Sr 407.771 Axial	5
Cr 266.342 Axial	1000	Sr 346.445 Axial	500
Cu 324.754 Axial	250	Ti 334.941 Axial	60
Fe 238.204 Axial	30	Ti 334.941 Radial	750
Fe 240.489 Radial	2500	V 268.796 Axial	250
K 766.491 Axial	30	V 290.644 Axial	1000
K 766.491 Radial	7500	Zn 213.857 Axial	60
Li 670.783 Axial	15	Zn 334.502 Axial	10000
Li 610.365 Radial	2500	Zr 343.823 Axial	125
Mg 279.553 Axial	7	Zr 327.927 Axial	500
Mg 279.800 Axial	1000		

Early Maintenance Feedback

Analyzing complex sample types such as wastewaters can be tough on the sample introduction system of an ICP-OES over extended analytical runs. The 5800 instrument includes a feature called Early Maintenance Feedback (EMF) that allows alerts to be set for instrument usage-based metrics (*6*). Various alerts can be set to remind analysts when to clean sample introduction components, replace pump tubing, clean/change pre-optics windows, clean the switching valve, and perform a wavelength calibration (Figure 2). Alerting analysts based on use, in contrast to set time intervals, means that maintenance is carried out only when needed.

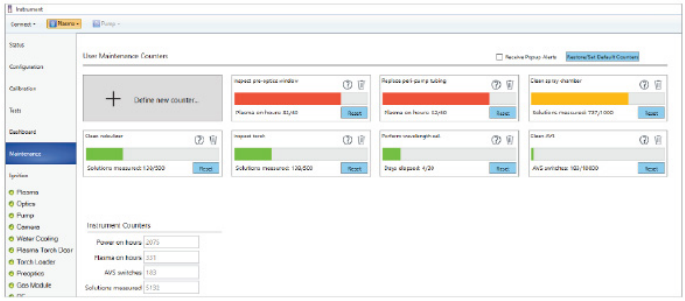

Figure 2. The EMF system monitors critical instrument parameters to maintain optimum analytical performance and reduce sample remeasurement.

Background correction

The ICP Expert software includes easy-to-use background correction techniques including Fitted Background Correction (FBC), which is suited to background correction of both simple and complex backgrounds (*7*). Since interferences are likely to arise from background structures during the analysis of wastewater samples, FBC was used in this study to ensure more accurate measurements. The combination of the FBC algorithm and Vista Chip III detector accurately corrects background structures without analyst intervention or method development. All wavelengths were corrected using FBC. An example of automatic background fitting for P 214.914 nm using FBC is shown in Figure 3.

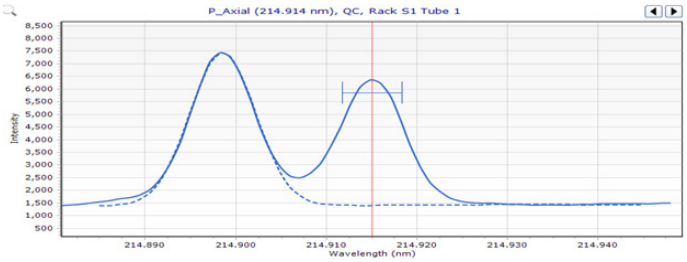

Figure 3. FBC automatically corrects for the nearby Cu peak to the left of P 214.914 (axial).

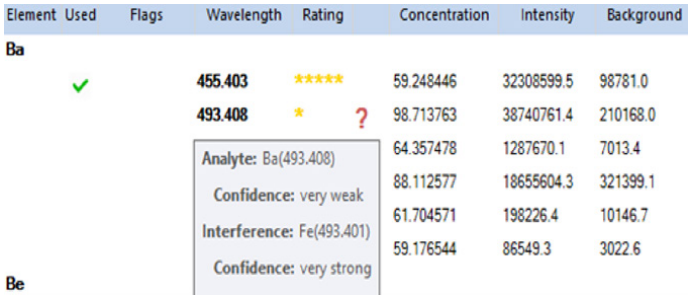

Figure 4. IntelliQuant can recommend the best wavelength to report for the quantitative analysis, as well as providing information why you should avoid certain wavelengths.

IntelliQuant

The IntelliQuant function of ICP Expert software is a fast semiquantitative data acquisition routine for ICP-OES. IntelliQuant collects data across the entire spectral range from 167 to 785 nm for every sample, providing a comprehensive data set. When performed as part of a quantitative method, an IntelliQuant scan provides valuable extra information about the sample or method. For example, IntelliQuant recommends the optimal wavelength to use in the final report using an automatic, software-assigned star-rating system for each wavelength.

In Figure 4, Ba 455.403 nm has received a 5-star ranking, while Ba 493.408 nm only received a 1-star ranking. Hovering over the "?" provides the user for the reasoning behind the poor star ranking for Ba 493.408 nm. In this example, Fe 493.401 nm has interfered on Ba 493.408 nm. Therefore, it is recommended this line is not reported in the quantitative analysis. Finally, Ba 455.403 nm has been assigned a green tick, meaning it has been ranked as the optimal wavelength to report for Ba.

Results and discussion

Method Detection Limits (MDLs)

The MDLs were calculated based on three sigma of 10 replicate measurements of the method blank. The results in Table 6 are an average of six determinations, performed on two different 5800 ICP-OES instruments over two consecutive days.

CRMs and matrix spike (MS) recovery tests

The recovery results for CWW-TM-D and NCS ZC 76307 in Table 7 are based on the average of three analytical runs performed using the 5800 ICP-OES. All elements recovered within ±10% of the expected values.

To determine the accuracy of the measurement of those elements not included in either of the CRMs but required by HJ 776, spike recovery tests were performed. CWW-TM-D was spiked with appropriate concentrations of each of these elements post-digestion. All recoveries were within ±10% of the spiked level, as shown in Table 8. The results are the average of three analytical runs performed on one instrument.

Table 6. MDLs for all wavelengths.

Element, Wavelength (nm) and Viewing mode	MDL in solution (µg/L)	MDL in sample (µg/L)	HJ 776 Requirement (µg/L)	Element, Wavelength (nm) and Viewing mode	MDL in solution (µg/L)	MDL in sample (µg/L)	HJ 776 Requirement (µg/L)
Ag 328.068 Axial	0.588	1.19	20.0	Mn 259.372 Axial	0.0965	0.195	4.00
Al 396.152 Axial	1.99	4.02	9.00	Mn 260.568 Radial	2.27	4.59	
Al 237.312 Axial	11.8	23.8		Mo 202.032 Axial	0.741	1.50	20.0
As 188.980 Axial	3.64	7.34	200	Mo 277.539 Axial	2.13	4.31	
As 234.984 Axial	11.9	24.1		Na 588.995 Axial	10.7	21.7	30.0
B 249.772 Axial	0.571	1.15	10.0	Na 588.995 Radial	254	513	
Ba 455.403 Axial	0.0490	0.0990	2.00	Ni 221.648 Axial	0.602	1.22	7.00
Ba 389.178 Axial	1.42	2.87		Ni 231.096 Axial	3.64	7.36	
Be 234.861 Axial	0.0416	0.0840	8.00	P 214.914 Axial	5.79	11.7	40.0
Be 249.473 Axial	3.28	6.63		Pb 220.353 Axial	3.74	7.55	70.0
Bi 223.061 Axial	6.94	14.0	40.0	S 180.669 Axial	5.37	10.8	520
Ca 393.366 Axial	0.170	0.343	20.0	Sb 217.582 Axial	4.88	9.86	60.0
Ca 315.887 Axial	2.80	5.65		Se 196.026 Axial	7.80	15.8	30.0
Cd 214.439 Axial	0.109	0.219	5.00	Si 251.611 Axial	1.94	3.92	20.0
Cd 226.502 Radial	2.17	4.39		Si 185.005 Axial	3.49	7.06	
Co 238.892 Axial	0.613	1.24	10.0	Sn 189.925 Axial	1.36	2.74	40.0
Co 230.786 Axial	0.698	1.41		Sn 181.059 Axial	9.52	19.2	
Cr 205.560 Axial	0.518	1.05	30.0	Sr 407.771 Axial	0.0187	0.0379	10.0
Cr 266.342 Axial	3.63	7.32		Sr 346.445 Axial	2.61	5.28	
Cu 324.754 Axial	1.13	2.28	6.00	Ti 334.941 Axial	0.162	0.327	20.0
Fe 238.204 Axial	0.344	0.695	2.00	Ti 334.941 Radial	1.66	3.36	
Fe 240.489 Radial	10.1	20.3		V 268.796 Axial	0.868	1.75	10.0
K 766.491 Axial	5.47	11.1	50.0	V 290.644 Axial	2.17	4.39	
K 766.491 Radial	212	428		Zn 213.857 Axial	0.291	0.587	4.00
Li 670.783 Axial	1.46	2.96	9.00	Zn 334.502 Axial	13.7	27.6	
Li 610.365 Radial	22.3	45.0		Zr 343.823 Axial	0.507	1.02	10.0
Mg 279.553 Axial	0.0248	0.0501	3.00	Zr 327.927 Axial	1.13	2.28	
Mg 279.800 Axial	2.61	5.28					

Table 7. Recovery results for the analysis of two CRMs.

Element, Wavelength (nm) and Viewing mode	CWW-TM-D			NCS ZC 76307		
	Measured (mg/L)	Expected (mg/L)	Recovery (%)	Measured (mg/L)	Expected (mg/L)	Recovery (%)
Ag 328.068 Axial	0.238	0.250	95.3			
Al 396.152 Axial	0.979	1.00	97.9			
Al 237.312 Axial	0.958	1.00	95.8			
As 188.980 Axial	0.256	0.250	103			
As 234.984 Axial	0.238	0.250	95.3			
B 249.772 Axial	1.03	1.00	103			
Ba 455.403 Axial	0.932	1.00	93.2			
Ba 389.178 Axial	0.953	1.00	95.3			
Be 234.861 Axial	0.232	0.250	93.0			
Be 249.473 Axial	0.232	0.250	92.9			
Cd 214.439 Axial	0.230	0.250	92.2	0.1049	0.1055	99.49
Cd 226.502 Radial	0.231	0.250	92.2	0.1037	0.1055	98.36
Co 238.892 Axial	0.918	1.00	91.8			
Co 230.786 Axial	0.971	1.00	97.1			
Cr 205.560 Axial	0.962	1.00	96.2	0.5592	0.5232	106.9
Cr 266.342 Axial	0.989	1.00	98.9	0.5478	0.5232	104.7
Cu 324.754 Axial	0.938	1.00	93.8	1.086	1.034	105.0
Fe 238.204 Axial	0.960	1.00	96.0			
Fe 240.489 Radial	1.02	1.00	102			
Mn 259.372 Axial	0.956	1.00	95.6			
Mn 260.568 Radial	0.994	1.00	99.4			
Mo 202.032 Axial	0.936	1.00	93.6			
Mo 277.539 Axial	0.970	1.00	97.0			
Ni 221.648 Axial	0.931	1.00	93.1	0.5288	0.5232	101.1
Ni 231.096 Axial	0.954	1.00	95.4	0.5447	0.5232	104.1
Pb 220.353 Axial	1.02	1.00	102	1.101	1.044	105.4
Sb 217.582 Axial	0.261	0.250	104			
Se 196.026 Axial	0.251	0.250	100			
Sr 407.771 Axial	0.990	1.00	99.0			
Sr 346.445 Axial	1.04	1.00	104			
V 268.796 Axial	0.979	1.00	97.9			
V 290.644 Axial	0.995	1.00	99.5			
Zn 213.857 Axial	0.963	1.00	96.3	5.600	5.212	1.074
Zn 334.502 Axial	0.916	1.00	91.6	5.466	5.212	1.049

Table 8. Recoveries for matrix spikes of CWW-TM-D CRM.

Element, Wavelength (nm) and Viewing Mode	Measured Sample (mg/L)	Spiked Concentration (mg/L)	Measured Spiked Sample (mg/L)	Recovery (%)
Bi 223.061 Axial	<MDL	0.485	0.481	99.2
Ca 393.366 Axial	0.00701	0.507	0.519	101
Ca 315.887 Axial	0.00727	0.511	0.523	101
K 766.491 Axial	<MDL	19.5	19.8	101
K 766.491 Radial	<MDL	18.5	18.7	101
Li 670.783 Axial	0.00160	14.9	15.1	101
Li 610.365 Radial	<MDL	13.5	13.6	101
Mg 279.553 Axial	0.00652	0.491	0.496	99.7
Mg 279.800 Axial	<MDL	0.496	0.500	101
Na 588.995 Axial	<MDL	19.5	19.6	101
Na 588.995 Radial	<MDL	20.1	20.4	101
P 214.914 Axial	0.0182	0.495	0.501	97.6
S 180.669 Axial	<MDL	0.525	0.529	101
Si 251.611 Axial	0.140	0.514	0.661	102
Si 185.005 Axial	0.133	0.508	0.659	104
Sn 189.925 Axial	<MDL	0.475	0.474	99.7
Sn 181.059 Axial	<MDL	0.491	0.486	99.1
Ti 334.941 Axial	0.0365	0.491	0.529	100
Ti 334.941 Radial	0.0358	0.479	0.520	101
Zr 343.823 Axial	0.00444	0.484	0.498	102
Zr 327.927 Axial	0.00307	0.507	0.524	103

Long-term stability

To assess the stability of the 5800 VDV ICP-OES, a 7.5 hour run was completed without recalibrating the instrument. Measurements of lab-made QCs were measured after the measurement of 10 household wastewater samples. Figure 5 shows the recoveries for all elements from the 35 QC measurements. All results were within 10% of the first QC reading and the %RSDs were below 2.3% for all wavelengths.

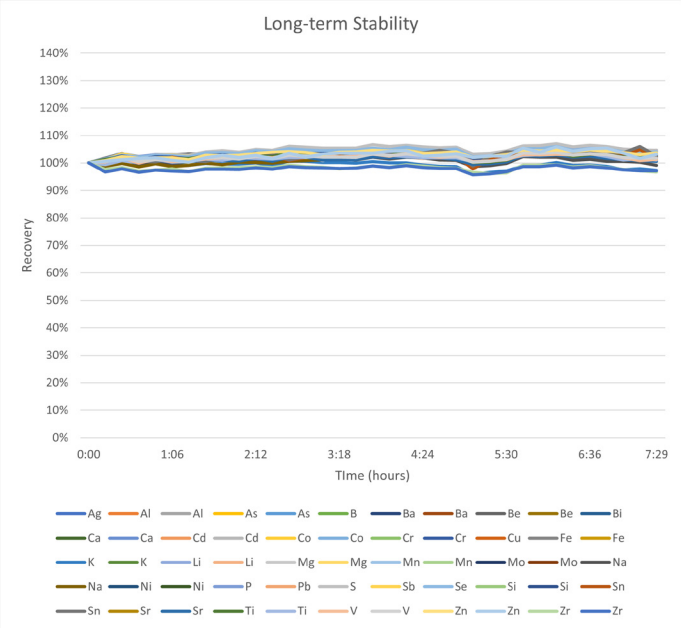

Figure 5. Long-term stability showing recovery of a QC sample analyzed after every 10 samples over seven hours.

Neb Alert

High-matrix samples, like wastewaters, can cause the deposition of crystalline particles onto components of the sample introduction system of the ICP-OES, especially the nebulizer. Partial or complete blockage of the nebulizer will lead to an inconsistent flow of the sample to the plasma, negatively affecting the accuracy and precision of the data. To prevent having to remeasure samples due to a nebulizer blockage, the "Neb Alert" feature in 5800 and 5900 ICP-OES instruments uses smart sensors that monitor the nebulizer backpressure during analysis (Figure 6). The analyst is alerted if there is a potential blockage, so that the nebulizer can be cleaned, rather than continuing and collecting potentially incorrect data. Leakage from a nebulizer gas line also triggers an alert within Neb Alert.

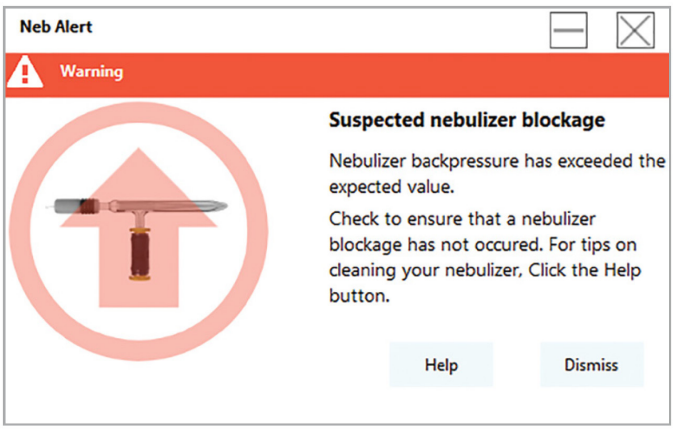

Figure 6. The Neb Alert feature triggers a warning if nebulizer backpressure exceeds the expected value.

Conclusion

The study has shown the effectiveness of the Agilent 5800 VDV ICP-OES with an AVS 7 switching valve for the analysis of various types of waters in accordance with the Chinese National Standard HJ 776. The AVS 7 improves productivity by reducing maintenance requirements on the VDV torch and reducing sample-to-sample measurement time down to 57 s.

The 5800 ICP-OES met performance requirements outlined in HJ 776-2015. The combination of the Vista Chip III detector and MultiCal software ensured that the 5800 met the linear dynamic range requirements for all elements. MDLs for all elements were below the MDLs specified in HJ 776 method, and good accuracy was demonstrated by the excellent recoveries of the CRMs and matrix spikes. The 5800 also maintained excellent stability throughout the 7.5 hour QC recovery test.

Smart software tools that were developed for the ICP Expert software ensure reliable, high-quality 5800 ICP-OES results for wastewater samples:

- MultiCal allows operators to use multiple calibrations for a single element, ensuring no compromise is made on collecting accurate low-level measurements and extending the dynamic range to high concentrations.
- To maximize instrument performance and minimize unplanned downtime, the EMF function was used to generate alerts for maintenance tasks based on the number of samples run.
- IntelliQuant was used to recommend/confirm the optimal wavelength to include in the final quantitative analysis report based on a star ranking of each wavelength.
- Analysts can be alerted to any issues relating to the nebulizer with the aid of smart sensors that monitor nebulizer backpressure. This Neb Alert feature is useful for the analysis of high matrix samples, such as wastewaters, and can save time remeasuring samples.

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