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ICP-OES

Nitrogen purge for the Thermo Scientific iCAP PRO Series ICP-OES

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Keywords

ICP-OES, purge gas, IDL, stability, cost efficiency, nitrogen

Goal

To demonstrate that nitrogen (N_2) can be used as a purge gas for the Thermo Scientific™ iCAP™ PRO Series ICP-OES without compromising analytical performance and show that instrument detection limits (IDL) and long-term stability of the analysis are comparable to using argon as a purge gas

Introduction

One of the major contributors to the operating costs for an ICP-OES instrument is the consumption of argon gas. Typically, total gas consumption of an instrument is divided into two categories: gas used to generate and sustain the plasma, and gas used to purge the optical system to remove atmospheric gases, which would lead to self-absorption specifically in the UV range of the spectrum. All commercially available ICP-OES instruments use argon to generate the plasma, and the majority also use argon for the purge gas, as it is perceived to produce excellent figures of merit. However, a more cost-effective purge gas would be nitrogen, and this should be considered if on-going running costs of the instrument are of concern. However, a lack of performance data to support the use of a nitrogen purge means many laboratory users are still hesitant to use nitrogen. This note sets out to demonstrate that nitrogen can successfully replace argon as a purge gas in the iCAP PRO Series ICP-OES without impacting analytical performance.

The iCAP PRO Series ICP-OES includes a unique optical system that was developed to ensure efficient purging. The optical system is housed in a sealed enclosure, which has the smallest feasible volume and incorporates the latest in sealing technology, reducing the amount of purge gas required. While in standby mode, the iCAP PRO Series ICP-OES requires minimal usage of 0.2 $L{\cdot}min^{-1}$ of purge gas to remove atmospheric gases from the optical system. This continued purge ensures fast startup times when the plasma is ignited, reducing the amount of gas used when the instrument is warming up.

Experiment

The instrument detection limit (IDL) and long-term stability can be used as indicators of the efficiency of the purged gas in ICP-OES. In this technical note, both will be assessed for nitrogen and argon purge using the iCAP-PRO Series ICP-OES. The instrumental conditions and parameters were the same for both argon and nitrogen purge (Table 1). The analysis mode was set to aqueous (axial and radial views) in all experiments.

Table 1. Instrument parameters

Parameter	Setting
Pump tubing	Sample: Tygon™ (orange/white) Drain: Tygon™ (white/white)
Pump speed	45 rpm
Spray chamber	Glass cyclonic spray chamber
Nebulizer	Glass concentric nebulizer
Nebulizer gas flow	0.55 $L{\cdot}min^{-1}$
Auxiliary gas flow	0.5 $L{\cdot}min^{-1}$
Coolant gas flow	12.5 $L{\cdot}min^{-1}$
Center tube	2.0 mm (quartz)
Torch	Thermo Scientific™ Enhanced Matrix Tolerance (EMT) Duo Torch
RF power	1,150 W
Radial viewing height	10 mm
Repeats	3
Exposure time	10 s (both iFR and eUV)
Uptake time	45 s
Wash time	30 s

Instrument detection limit (IDL)

All models of the iCAP PRO Series ICP-OES offer analysis in the iFR (intelligent Full Range) mode. However, the Thermo Scientific iCAP PRO XP ICP-OES and Thermo Scientific iCAP PRO XPS ICP-OES models also offer an additional eUV mode (enhanced ultraviolet). The iFR mode allows complete analysis of the wavelength range (167–852 nm) of the iCAP PRO Series ICP-OES, enabling simultaneous measurements of all elements in the analysis. The eUV mode enables extra sensitivity for elements emitting light in the UV region of the spectrum (167–240 nm). To demonstrate both modes (iFR and eUV) and both plasma views (axial and radial), the IDL experiments were designed and carried out as detailed below. Thirty elements were analyzed for the IDL tests (Table 4). These elements and wavelengths cover the wavelength range of the instrument and represent typical elements analysis. Calibration standards were prepared with 0.2% HNO_3 (trace metal grade) to final concentrations of 5, 20, 50, and 100 $\mu g{\cdot}L^{-1}$. Each of the blank solutions were prepared using 0.2% HNO_3 (trace metal grade) and measured with 10 replicates (3 blanks in total) after plasma ignition and instrument stabilization. For each measurement, a 10-second exposure time was applied with both axial (iFR and eUV) and radial views (iFR and eUV). The IDL tests were performed on 3 consecutive days and the average IDL was calculated. The experiment was carried out with both argon and nitrogen purges. The same instrument was used for all analyses.

IDL was derived from the raw intensity data of the calibration standards and blanks using the following equation:

$$IDL = 3SD_{blk} \frac{STD_{conc}}{STD_x - BLK_x}$$

Where:

IDL is the instrument detection limit

SD_{blk} is the standard deviation of the intensities of the multiple blank measurements

STD_{conc} is the concentration of the standard

STD_x is the mean signal for the standard

BLK_x is the mean signal for the blank

The three-fold multiplier is based on the student's *t*-test table and indicates that the detection limit is calculated using a confidence range of 99%.

Long-term stability

The long-term stability tests were carried out by spiking a tap water sample (500 mL) with the 50 $\mu g{\cdot}L^{-1}$ single element standards of Al, As, Cd, Cr, Co, Mn, Pb, Tl, and V. The spiked samples were analyzed for the spiked elements as well as for the elements Ba, Cu, K, Mg, Na, S, Si, Sr, and Zn, which occur naturally in the tap water in concentrations above the respective limit of quantifications (3 × LOD). An internal standard (10 $mg{\cdot}L^{-1}$ of yttrium) was used. The instrument was calibrated prior to all long-term stability tests with the calibration standard concentrations similar to the IDL test. The tests used the parameters in Table 1 and were carried out as follows:

1. The instrument was connected to the select purge gas, either argon or nitrogen.
2. Initial purge took place in standby mode overnight using 0.2 $L{\cdot}min^{-1}$ for the optical purge (to simulate an instrument installed in a typical laboratory).
3. From standby, the plasma was ignited and allowed to stabilize for 15 minutes. During this time the instrument was purged under normal operating conditions of 2.2 $L{\cdot}min^{-1}$ optical purge.
4. The analysis took place over a 12-hour period.
5. Percent recovery was then calculated from normalized concentrations, i.e., the ratio of each data point (average concentration) to the overall average concentration measured over a 12-hour period. The overall concentration of each element was set as 100% recovery, and each element's average concentration was normalized and plotted against time (Figures 1 and 2).

Argon and nitrogen gas used in the experiments met all parameters specified in Table 2.

Table 2. Purge gas specification for iCAP PRO Series ICP-OES

Purge gas specification	Argon (Ar)	Nitrogen (N_2)
Purity	≥99.995%	≥99.995%
Oxygen content	<10 ppm	<10 ppm
Water content	<10 ppm	<10 ppm
Max supply rate	10.2 $L{\cdot}min^{-1}$	10.2 $L{\cdot}min^{-1}$
Operating pressure	0.55–0.6 MPa (5.5–6 bar)	0.55–0.6 MPa (5.5–6 bar)

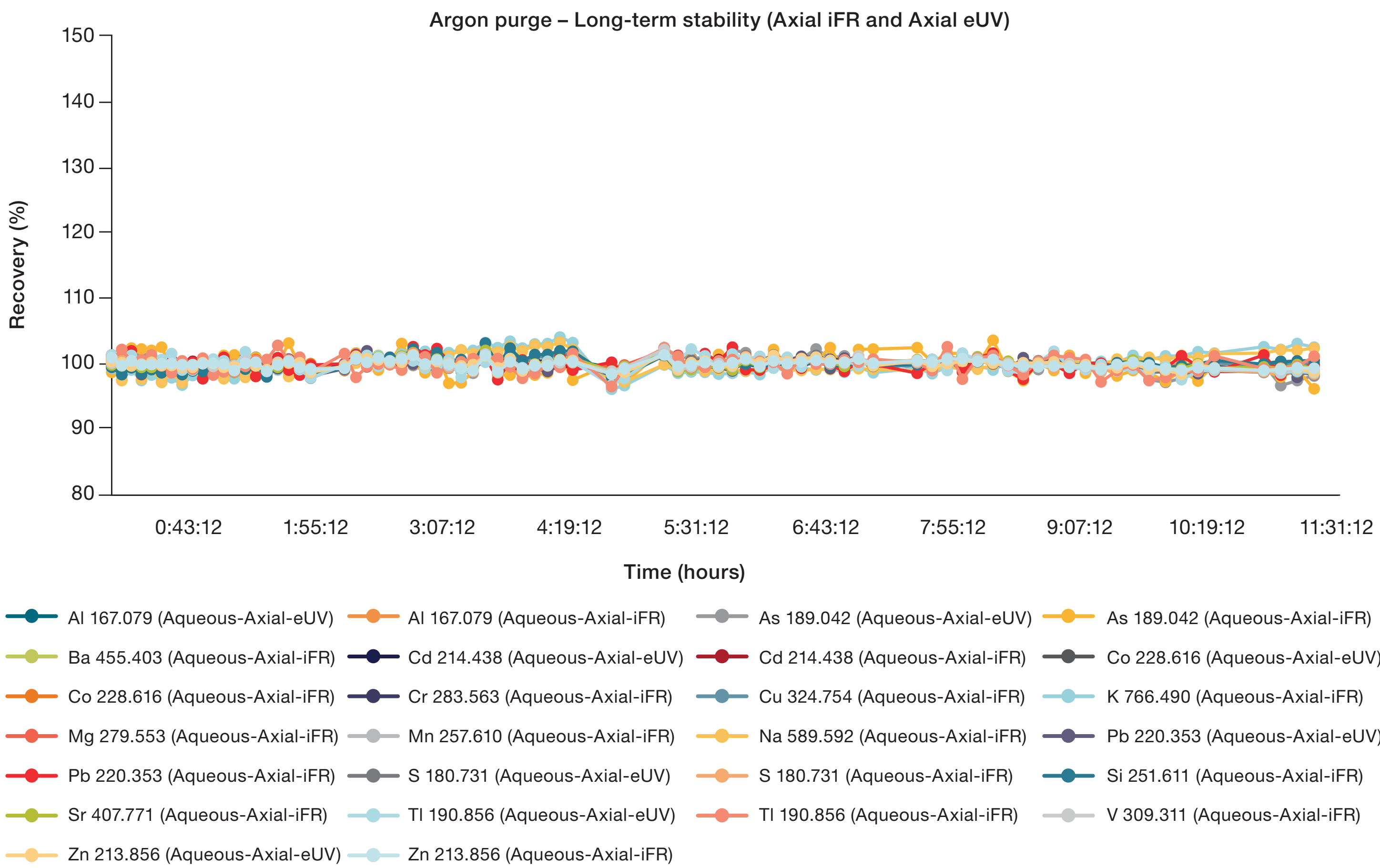

Figure 1. Long-term stability of elements listed in Table 1 with 15 min argon purge. 100% recovery refers to an overall average concentration over a 12-hour period.

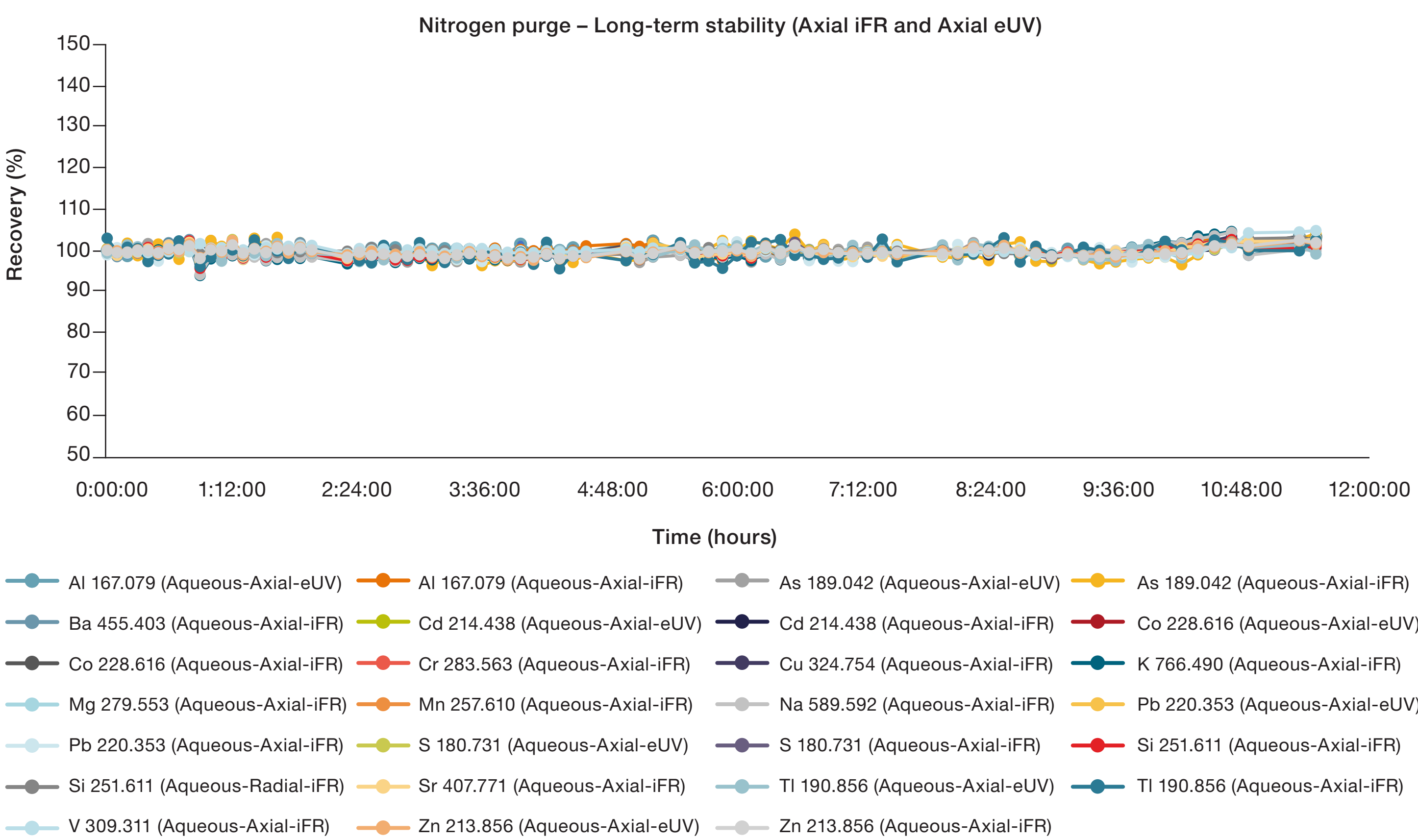

Figure 2. Long-term stability of elements listed in Table 1 with 15 min nitrogen purge. 100% recovery refers to an overall average concentration over a 12-hour period.

In addition to the standard purge modes, the iCAP PRO XP ICP-OES and the iCAP PRO XPS ICP-OES systems both have a boost purge feature that enables faster startup. To examine the measurement stability of the boost purge, an additional test was performed for the 2-hour short-term stability test with a nitrogen purge using the iCAP PRO XPS ICP-OES.

The iCAP Series Multi-Element Test Solution (METS) was used as a sample for this short-term stability test. The list of elements included in the METS can be found in Table 3.

Table 3. Composition of the iCAP PRO METS

Concentration	Element(s)
10 mg·L^{-1}	P
5 mg·L^{-1}	K, Ni
1 mg·L^{-1}	Al, Cu, Mn
0.2 mg·L^{-1}	Ba, Ca, Mg, Zn

In this experiment, the instrument conditions, analysis mode, and parameters were the same as those used in the long-term stability and IDL tests described earlier. However, the instrument was not calibrated prior to the tests. The normalized intensity data was used for the 2-hour stability plot (Figure 3). The 2-hour stability tests were carried out as follows:

1. The instrument (iCAP PRO XPS ICP-OES) was connected to the nitrogen gas for purging.
2. The initial purge took place in standby mode overnight similar to the long-term stability tests.
3. Five minutes of boost purge was performed and thereafter the plasma was ignited.
4. After completion of the purge, the analysis took place immediately and was carried out over a 2-hour period.
5. Percent recovery was calculated from the normalized intensity in the same manner as in the 12-hour long-term stability tests, i.e., the overall average intensity (2-hour period) of each element was set as 100% recovery, and each element's average intensity was normalized by the overall average intensity. Later, the normalized data was plotted against time (Figure 3).

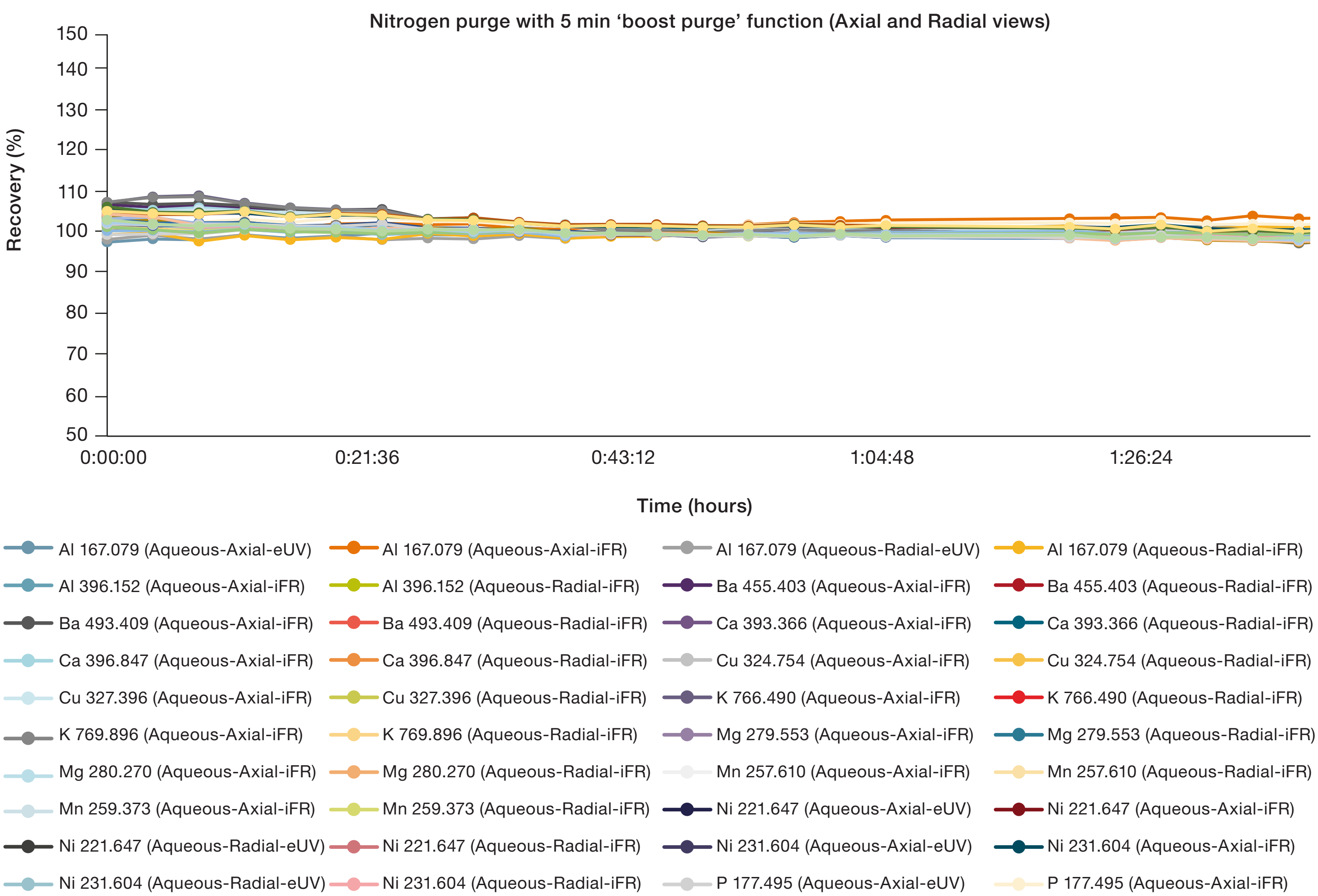

Figure 3. Short-term stability (~2 h) of elements listed in Table 3 with 5 min nitrogen boost purge. 100% recovery refers to an overall average concentration over a 2-hour period.

Results and discussion

Instrument Detection Limit (IDL)

The comparison of average IDLs between the nitrogen and argon purge are listed in Table 4. Overall, the average IDLs of these 30 selected elements from the nitrogen purge are in a similar range as the average IDLs obtained from the argon purge.

Table 4. The typical detection limits for the iCAP PRO Series ICP-OES using argon and nitrogen as a purge gas

Element	Wavelength	Axial (iFR mode – µg·L^{-1})		Axial (eUV mode – µg·L^{-1})		Radial (iFR mode – µg·L^{-1})		Radial (eUV mode – µg·L^{-1})	
	(nm)	Argon average	Nitrogen average	Argon average	Nitrogen average	Argon average	Nitrogen average	Argon average	Nitrogen average
Ag	328.068	0.582	0.316	–	–	1.33	1.089	–	–
Al	167.079	0.172	0.144	0.058	0.079	0.794	0.546	0.518	0.342
As	189.042	2.69	3.61	1.84	1.76	8.42	12.2	5.91	6.08
B	249.773	0.805	0.388	–	–	0.898	1.074	–	–
Ba	455.403	0.040	0.021	–	–	0.112	0.086	–	–
Be	313.107	0.028	0.022	–	–	0.089	0.073	–	–
Cd	214.438	0.104	0.145	0.079	0.084	0.633	0.587	0.406	0.398
Co	228.616	0.557	0.572	0.379	0.322	2.39	2.07	1.60	1.54
Cr	283.563	0.347	0.319	–	–	1.06	0.807	–	–
Cu	324.754	0.513	0.344	–	–	1.66	1.26	–	–
Fe	238.204	0.347	0.301	0.284	0.243	1.59	1.20	0.691	–
Hg	184.950	0.770	0.683	0.436	0.501	2.00	2.40	1.32	1.61
K	766.490	1.057	0.991	–	–	18.0	12.5	–	–
Mg	279.553	0.044	0.174	–	–	0.029	0.061	–	–
Mn	257.610	0.065	0.046	–	–	0.209	0.144	–	–
Mo	202.030	0.678	0.702	0.445	0.366	2.735	2.51	1.53	0.933
Na	589.592	1.152	1.13	–	–	13.3	5.14	–	–
Ni	221.647	0.467	0.568	0.252	0.252	1.813	1.56	1.35	1.14
P	177.495	1.41	2.28	1.36	1.39	8.57	6.64	5.38	5.14
Pb	220.353	2.08	2.26	1.36	0.851	8.70	10.7	5.53	5.91
S	180.731	3.02	3.02	2.45	2.32	9.81	13.6	5.52	6.10
Sb	217.581	5.09	3.64	3.35	2.75	13.8	11.0	11.4	7.26
Se	196.090	4.76	3.04	2.70	2.73	15.1	12.5	8.22	9.45
Si	251.611	1.18	1.26	–	–	3.76	2.85	–	–
Sn	189.989	2.26	1.33	1.47	0.828	6.38	8.01	9.22	3.86
Sr	407.771	0.037	0.025	–	–	0.037	0.054	–	–
Ti	323.452	0.219	0.221	–	–	0.892	0.831	–	–
Tl	190.856	2.87	2.80	1.34	2.10	19.3	15.1	11.8	11.5
V	309.311	0.667	0.684	–	–	1.021	0.700	–	–
Zn	213.856	0.253	0.227	0.121	0.129	0.483	0.708	0.323	0.414

Long-term stability

The analysis of the 18 elements in the spiked tap water sample exhibited excellent long-term stability with both argon and nitrogen purge (Figures 1 and 2). The nitrogen purge provided excellent long-term stability for all analytes with less than ±10% variation of the recoveries. The stability of the analytical run with a nitrogen was comparable to the argon purge. The consistent recovery and stability of all analytes from the nitrogen purge confirmed the suitability of nitrogen as a purge gas for long-term analysis.

For the iCAP PRO XP ICP-OES and iCAP PRO XPS ICP-OES systems coming from Standby, the use of the boost purge for 5 minutes is sufficient to achieve very good measurement stability within ±10% (Figure 3). It is recommended that a 15-minute plasma warm-up is performed prior to the analysis.

Conclusion

All of the iCAP PRO Series ICP-OES systems offer the nitrogen purge inlet for a purge gas option. In terms of instrument detection limits and long-term stability, there are no significant differences between using nitrogen or argon as a purge gas for the iCAP PRO Series ICP-OES systems. For the iCAP PRO XP ICP-OES and the iCAP PRO XPS ICP-OES systems, a 5-minute boost purge is adequate to offer measurements within an acceptable range of stability. Replacing argon with a nitrogen purge can save up to 40% of purge gas daily expense (based on UK prices in July 2022). The use of a nitrogen purge gas with the iCAP PRO Series ICP-OES systems is an effective solution for laboratories seeking to reduce the ongoing running cost of trace element analysis.

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