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Comprehensive and Fast Multi-Elemental Analysis Using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

Presented by: Mike Mourgas Applications Scientist Trace Elemental Analysis

WS73686-EN 0720S

The world leader in serving science

A Complete Portfolio of Elemental Analysis Solutions

Features include:

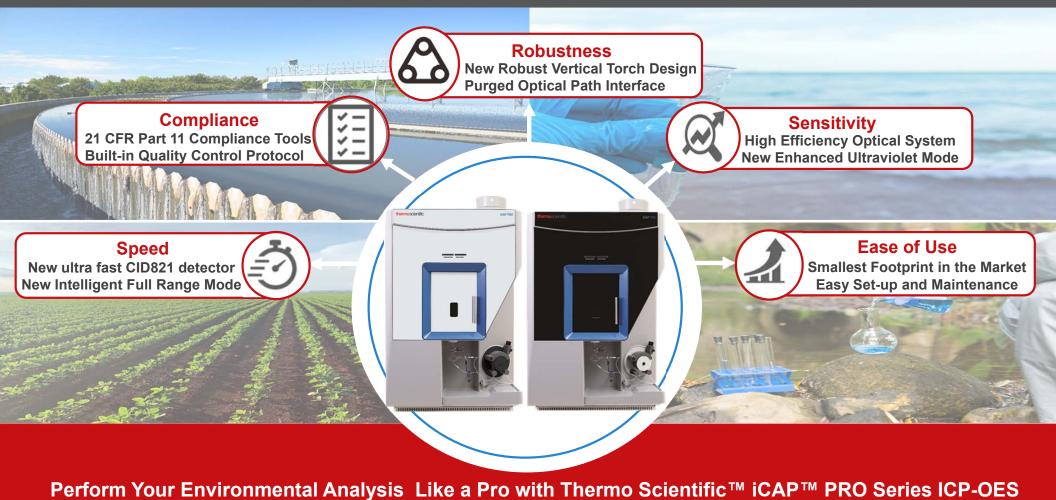
- Single or multi-element analysis
- Low-cost, high-throughput systems
- Space-saving design
- Low gas consumption



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Introducing the New Thermo Scientific iCAP PRO Series ICP-OES



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Thermo Scientific iCAP PRO Series ICP-OES: Solutions for Every Environmental Sample



Four New Models Optimized for Performance, Flexibility and Speed

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Thermo Scientific iCAP PRO Series ICP-OES – Core Technologies



Full Frame imaging



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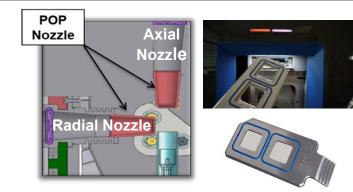
Vertical Torch and POP Interface Designed for Enhanced Robustness



Vertical Torch & Inner Torch Box

- Vertical torch for all models
- Adjustable radial viewing height for radial and duo instruments
- Removable inner torch box Designed for corrosion resistance, easy to remove and clean when needed





Optimized Exhaust Flow

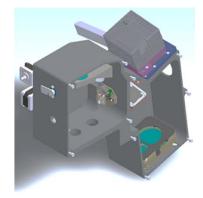
- Proprietary exhaust flow design through torch box ensures:
 - Optimal cooling of torch
 - · Stability of the plasma
 - Minimal deposition of the torch and center tube

POP interface and window

- Durable ceramic cones Purge gas exits from optical system through cones to remove interferences that can compromise sensitivity
- New POP window Protects fore optics from dust, dirt and contamination from the plasma, easy to remove and clean when needed



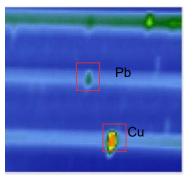
Optical System and New CID Detector for Enhanced Speed, Sensitivity and Robustness



Improved speed and sensitivity

7

- **Simultaneous** measurement of the whole spectrum in one acquisition!
- Full wavelength coverage Range of 167-852 nm using iFR Mode or 167-240 nm with eUV mode
- **High quality optics** Maximum light throughput to maintain speed





- Greater order separation Reduced order overlap and interferences (Pb in 10,000 ppm Cu)
- Resolution <7pm at 200 nm
- Improved stability Optimized air flows to prevent sample deposition



Robust optics

- Mirror protection Beam blocker to prevent extreme UV exposure
- Sealed optics Moving parts outside the optical tank
- Efficient purge Just 3.4 L/min



iCAP PRO Series ICP-OES Detector

New Camera – Thermo Scientific CID821

Large chip allows simultaneous readout Array size of 2048 x 2048 pixels

Improved speed

Pre-exposure not required Ultra-fast signal readout, 30-40% faster than previous generation detector

Reduced interferences

Maximum order separation (e.g., Cu on Pb) Anti-blooming advantage over CCD detectors Off-axis window eliminates optical scatter





iCAP 7000 Series ICP-OES CID86 iCAP PRO Series ICP-OES CID821



Features for Enhanced Ease of Use



Door Closed

Door Open

New Features

- New Torch Box Door Sliding door for easy access to the torch interface
- New LED instrument status panel Allows user to know status of the instrument or analysis from across the laboratory



Small Footprint and Easy Installation

- Smallest footprint on the market Dimension: 24.2"x27.2"x36.7" (LxWxH), optimizes laboratory bench space
- Easy access Improved connection area, can be pushed close against the wall





Sample Introduction System

- Sample introduction kits Five application specific kits
- Clip-in sample introduction system – easy to assemble/disassemble
- Ceramic torch for enhanced robustness when analyzing high matrix sample continuously over long periods



Improved Speed – Reduced Warm-Up Time

Reduced warm-up time

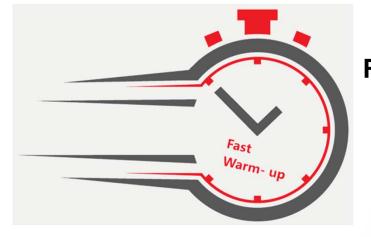
- Efficiency Helps users to increase their lab efficiency
- Speed Fast warm-up with system stability and quick purge
- Software Software stabilization enables fast measurements <u>from</u> <u>standby</u>

intelligent Full Range - iFR

- **Standard purge** Fast warm-up from system standby
- Quick start up Just five minutes from standby to first measurement

enhanced UV - eUV

- Standard purge for UV No special requirements from standby (gas on)
- Enhanced purge from power off Enhanced UV, fast warm-up from power off (no power, no gas)



Ready to go and use much faster than iCAP 7000 Series ICP-OES

Increase your laboratory efficiency

Applies to the Thermo Scientific[™] iCAP[™] PRO XP ICP-OES and the Thermo Scientific[™] iCAP[™] PRO XPS ICP-OES

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Thermo Scientific iCAP PRO Series ICP-OES Software

Thermo Scientific[™] Qtegra[™] Intelligent Scientific Data Solution[™] (ISDS) Software

- Benefits of the Qtegra ISDS Software
 - Installed on over > 6000 instruments
 - Intuitive, streamline workflow platform
 - Continues to support existing liquid sampling accessories
 - A range of new features added for ease of use
 - 21 CFR Part 11 compliance tool set
 - Same software platform as the Thermo Scientific ICP-MS instruments for easy cross-training and operation between techniques





Method Validation – A Must for Confirming Method Optimization and Data Quality

- Method Validation
 - Required to ensure that the method optimized results in accurate, precise and reproducible results.

Method validation achieved through:

- MDL or IDL determinations DLs determined for the instrument and each sample method
- Calibration curve correlation coefficient (R²) of at least 0.995, the closer to 1.0 the better
- Linear Dynamic Range (LDR) determination
- Precision tests measured by %RSD between 3 sample replicates (short term precision)
- Accuracy tests matrix spiking of samples and analysis of a Certified Reference Material (CRM)
- Repeat above tests over several days (long term precision and accuracy)
- Quality Control Protocol built in the Qtegra ISDS Software
 - For routine, continuous monitoring of the analysis
 - Includes automatic actions that can be selected upon failure of a QC standard





Let Us Look at Specific Applications

Application Data for the iCAP PRO Series ICP-OES

- Analysis of water samples according to USEPA Method 200.7
 using the iCAP PRO XPR ICP-OES
- 25% NaCl measurements using the iCAP PRO XP Duo vs. Radial



Analysis of Water Samples by US EPA Method 200.7

Analysis of water samples by USEPA Method 200.7 using the iCAP PRO XPS ICP-OES



- This note describes the analysis of water samples by Method 200.7
- Challenge:
 - Compliance with comprehensive method quality control protocol

National Primary Drinking Water Regulations						
Contaminant	MCL (mg/L)	MCLG (mg/L)	Contaminant	MCL (mg/L)	MCLG (mg/L)	
Antimony	0.006	0.006	Copper	1.3	1.3	
Arsenic	0.01	0	Lead	0.015	0	
Barium	2.0	2.0	Mercury	0.002	0.002	
Beryllium	0.004	0.004	Selenium	0.05	0.05	
Cadmium	0.005	0.005	Thallium	0.002	0.002	
Chromium	0.1	0.1	Uranium	0.03	0	

National Secondary Drinking Water Regulations				
Contaminant	MCL (mg/L)			
Aluminum	0.05 - 0.2			
Copper	1			
Iron	0.3			
Manganese	0.05			
Silver	0.1			
Zinc	5			

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Analysis of Water Samples by US EPA Method 200.7

Instrumentation

- iCAP PRO XPS Duo ICP-OES
- Teledyne CETAC ASX-560 autosampler

Samples and Standards

- All calibration and QC standards prepared using 1000 mg/L stock standards in 1.5% HNO₃
- Internal Standard: 5 mg/L Yttrium added online
- Samples Drinking water, trench water and well water

Analysis

- MDL study
 - Reagent blank spiked at low concentrations
- LDR study
- Interference Study
 - Single element SIC solutions
- Run sequence Calibration QCS

IPC Check Blank 10 Samples IPC Check Blank 152 total samples, including QC and calibration standards

Parameter	Setting
Pump Tubing	Sample: Tygon® orange/white Drain: Tygon® white/white
Pump Speed	45 rpm
Spray Chamber	Glass Cyclonic
Nebulizer Gas Flow	Glass Concentric
Coolant Gas Flow	12 L/min
Auxiliary Gas Flow	0.5 L/min
Nebulizer Gas Flow	0.5 L/min
Center Tube	2 mm
RF Power	1150 W
Repeats	3
Radial view height	10 mm
Exposure Time	Axial View – 10 seconds Radial View – 10 seconds

*Analysis using iFR mode for all 31 elements



Analysis of Water Samples by US EPA Method 200.7 – Results

MDL and LDR Results

• MDL

 All results shown are below the levels for the National Primary and Secondary Drinking Water standards.

• LDR

• LDR results are more than sufficient for the analysis of typical water samples.

*UMCR 3 – Third Unregulated Contaminant Water Rule

Analyte	Wavelength (nm)	Plasma View	LDR (mg/L)	MDL (µg/L)	Level of Interest (µg/L)
Ag	328.608	Axial	>10	0.84	100
AI	308.215	Radial	>1000	21	50 – 200
As	193.759	Axial	>100	2.1	10
Ва	455.403	Axial	>2	0.47	2000
Be	234.861	Axial	>10	0.08	4
Cd	226.502	Axial	>10	0.25	5
Со	228.616	Axial	>10	0.75	1*
Cr	284.325	Axial	>10	0.29	100 / 0.2*
Cu	224.700	Axial	>10	0.51	1300
Fe	258.940	Radial	>1000	3.7	300
Mn	257.610	Axial	>10	0.06	50
Мо	203.844	Axial	>10	0.90	1*
Pb	220.353	Axial	>100	3.2	15
Sb	206.833	Axial	>100	3.3	5
Se	196.090	Axial	>100	4.8	50
TI	190.856	Axial	>10	0.50	0.2*
Zn	213.856	Axial	>2	0.02	5000



Analysis of Water Samples by US EPA Method 200.7 – Results

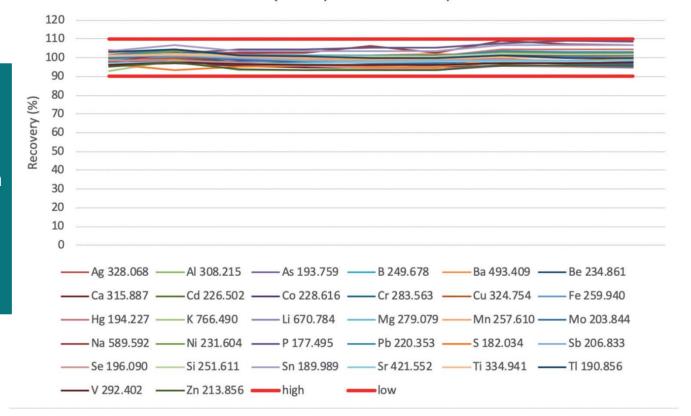
		Drinking Water			Trench Water		Well Water			
	Analyte	Unspiked (mg/L)	Spiked (mg/L)	Recovery (%)	Unspiked (mg/L)	Spiked (mg/L)	Recovery (%)	Unspiked (mg/L)	Spiked (mg/L)	Recovery (%)
	As	<mql< th=""><th>0.200</th><th>100.0</th><th><mql< th=""><th>0.203</th><th>101.5</th><th><mql< th=""><th>0.203</th><th>101.5</th></mql<></th></mql<></th></mql<>	0.200	100.0	<mql< th=""><th>0.203</th><th>101.5</th><th><mql< th=""><th>0.203</th><th>101.5</th></mql<></th></mql<>	0.203	101.5	<mql< th=""><th>0.203</th><th>101.5</th></mql<>	0.203	101.5
	Са	40.52	42.43	95.5	49.92	57.39	99.6	46.20	53.66	99.5
	Cd	<mql< th=""><th>0.199</th><th>99.5</th><th><mql< th=""><th>0.196</th><th>98.0</th><th>0.01</th><th>0.199</th><th>99.0</th></mql<></th></mql<>	0.199	99.5	<mql< th=""><th>0.196</th><th>98.0</th><th>0.01</th><th>0.199</th><th>99.0</th></mql<>	0.196	98.0	0.01	0.199	99.0
Access to Decutto	Cu	0.024	0.319	98.3	<mql< th=""><th>0.291</th><th>97.0</th><th>0.007</th><th>0.296</th><th>96.3</th></mql<>	0.291	97.0	0.007	0.296	96.3
Sample Results	Fe	0.045	0.239	97.0	1.360	8.701	97.9	27.40	34.82	98.9
	Hg	<mql< th=""><th>0.196</th><th>98.0</th><th><mql< th=""><th>0.196</th><th>98.1</th><th><mql< th=""><th>0.197</th><th>98.5</th></mql<></th></mql<></th></mql<>	0.196	98.0	<mql< th=""><th>0.196</th><th>98.1</th><th><mql< th=""><th>0.197</th><th>98.5</th></mql<></th></mql<>	0.196	98.1	<mql< th=""><th>0.197</th><th>98.5</th></mql<>	0.197	98.5
All spike	K	2.747	7.795	101.0	12.56	15.31	110.0	1.401	4.116	108.6
recoveries for the different water	Mg	4.271	11.60	97.7	7.863	14.95	9.45	6.953	14.02	94.2
samples were	Na	14.24	19.67	108.6	145.31	170.8	102.0	92.85	118.3	101.8
within the required	Р	0.015	1.644	108.6	0.102	1.730	108.5	1.185	2.742	103.8
recovery range of	Pb	<mql< th=""><th>0.197</th><th>98.5</th><th><mql< th=""><th>0.192</th><th>96.0</th><th>0.077</th><th>0.266</th><th>94.5</th></mql<></th></mql<>	0.197	98.5	<mql< th=""><th>0.192</th><th>96.0</th><th>0.077</th><th>0.266</th><th>94.5</th></mql<>	0.192	96.0	0.077	0.266	94.5
85 – 115%.	Sb	<mql< th=""><th>0.200</th><th>100.0</th><th><mql< th=""><th>0.195</th><th>97.5</th><th><mql< th=""><th>0.197</th><th>98.5</th></mql<></th></mql<></th></mql<>	0.200	100.0	<mql< th=""><th>0.195</th><th>97.5</th><th><mql< th=""><th>0.197</th><th>98.5</th></mql<></th></mql<>	0.195	97.5	<mql< th=""><th>0.197</th><th>98.5</th></mql<>	0.197	98.5
	Se	<mql< th=""><th>0.193</th><th>96.5</th><th><mql< th=""><th>0.193</th><th>96.5</th><th><mql< th=""><th>0.197</th><th>98.5</th></mql<></th></mql<></th></mql<>	0.193	96.5	<mql< th=""><th>0.193</th><th>96.5</th><th><mql< th=""><th>0.197</th><th>98.5</th></mql<></th></mql<>	0.193	96.5	<mql< th=""><th>0.197</th><th>98.5</th></mql<>	0.197	98.5
	SO4	40.43	43.82	113.2	77.69	93.13	103.1	1.295	16.80	103.5
	TI	<mql< th=""><th>0.198</th><th>99.0</th><th><mql< th=""><th>0.281</th><th>93.7</th><th><mql< th=""><th>0.283</th><th>94.3</th></mql<></th></mql<></th></mql<>	0.198	99.0	<mql< th=""><th>0.281</th><th>93.7</th><th><mql< th=""><th>0.283</th><th>94.3</th></mql<></th></mql<>	0.281	93.7	<mql< th=""><th>0.283</th><th>94.3</th></mql<>	0.283	94.3
	Zn	0.0009	0.22	109.6	0.0013	0.22	109.4	0.282	0.48	99.0

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Analysis of Water Samples by US EPA Method 200.7

Sample Results

- Stability demonstrated by the analysis of the Instrument Performance Check (IPC) Solution
- 9 IPC solutions analyzed over the run, all results within the 90 – 110% recovery as shown in the graph



Recovery of repeated IPC samples



Analysis of Water Samples by US EPA Method 200.7 – Summary

- Compliance with the comprehensive Method 200.7 QC protocol for the determination of 31 elements in various water samples was demonstrated using the iCAP PRO XPS Duo ICP-OES.
- The productivity tools of Qtegra ISDS Software combined with the speed of the iCAP PRO XPS ICP-OES using the iFR analysis mode resulted in an analysis time of <u>1 minute</u> and <u>58 seconds per sample, or 30 samples per hour.</u>
- Method 200.7 QC protocol was easily implemented in the LabBook using the built-in QC features of the Qtegra ISDS Software.
- The optimized vertical torch combined with the highresolution optics minimized physical and spectral interferences.

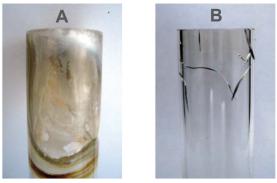




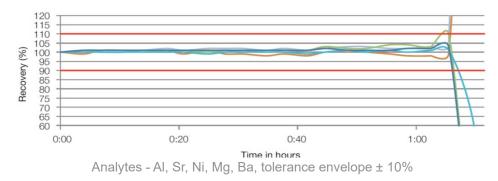
Analysis of 25% NaCl to Demonstrate High Matrix Tolerance

Challenges:

- Devitrification of quartz glass ICP torch
- Clogging of center tube due to salt deposition
- Clogging of nebulizer
- High RF power, high nebulizer gas flow, etc., required for high matrix samples
- Poor data stability or failure over long runs



(A) Devitrification and (B) premature failure of quartz glass torch

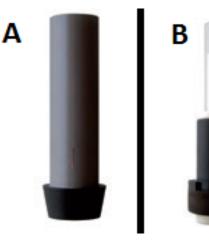


Poor stability/failure in analysis in less than 2 hrs. with normal set-up

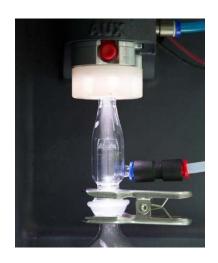
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Analysis of 25% NaCI – Sample Introduction and Instrumentation

- Sample introduction system
 - Burgener Mira Mist nebulizer
 - Baffled spray chamber
 - Sheath gas adaptor with additional gas supply
 - Ceramic D-torch
- iCAP PRO XP ICP-OES
 - Dedicated Radial traditional choice for high salt solutions
 - Duo New Vertical Torch design on the iCAP PRO Series ICP-OES



A. Ceramic torch outer tube
 B. Torch with quartz outer
 tube for displaying inner
 ceramic tube



Sheath gas adaptor



Analysis of 25% NaCl - Instrument Parameters

- Both configurations of the iCAP PRO XP ICP-OES were used for the analysis to compare results
- Additional gas is argon for the Sheath Gas Adaptor

Parameter	Setting for iCAP PRO XP ICP-OES Radial	Setting for iCAP PRO XP ICP-OES Duo		
Pump Tubing	Sample Tygon® orange/ white	Sample Tygon® orange/ white		
	Drain Tygon® white/white	Drain Tygon® white/white		
Spray Chamber	Baffled cyclonic	Baffled cyclonic		
Nebulizer	Burgener Mira Mist	Burgener Mira Mist		
Center Tube	2.0 mm (ceramic)	2.0 mm (ceramic)		
Torch	Ceramic D-Torch Radial	Ceramic D-Torch Duo		
Pump Speed	45 rpm	45 rpm		
Flush Pump Speed	100 rpm	100 rpm		
Pump Stabilization Time	10 s	10 s		
Wash Time	30 s	30 s		
Nebulizer Gas Flow	0.55 L∙min ⁻¹	0.55 L·min ⁻¹		
Auxiliary Gas Flow	0.5 L·min ⁻¹	1.5 L·min ⁻¹		
Coolant Gas Flow	12.0 L L·min ⁻¹	12.0 L·min ⁻¹		
Additional Gas 0.15 L L·min ⁻¹		0.15 L·min ⁻¹		
RF Power	1400 W	1350 W		
Radial Viewing Height	11 mm	11 mm		
Exposure Time	iFR 10 s	Radial iFR 10 s, Axial iFR 10 s		

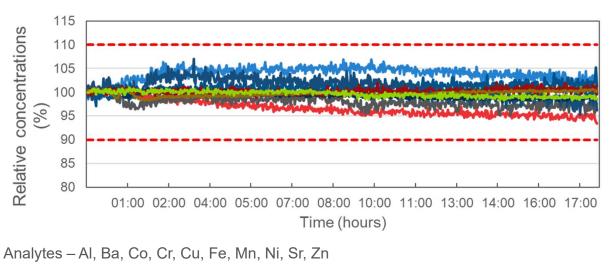


Results – Sensitivity and Long-Term Stability Experiment

Method Detection Limits (MDL) obtained from different configurations of the iCAP PRO XP ICP-OES

Element and wavelength (nm)	Radial only MDL (µg∙L⁻¹)	Duo – Radial view MDL (µg⋅L ^{₋1})	Duo – Axial view MDL (µg⋅L ⁻¹)
AI 167.079	2.33	7.46	4.91
Ba 455.403	1.17	1.68	0.75
Co 228.616	7.79	4.78	3.37
Cr 205.560	2.73	3.94	1.58
Cu 324.754	7.82	5.65	1.93
Fe 259.940	9.8	13.06	6.58
Mn 257.610	3.88	8.15	2.31
Ni 221.647	4.71	5.81	2.58
Sr 407.771	7.7	8.48	4.41
Zn 213.856	2.54	2.85	0.72

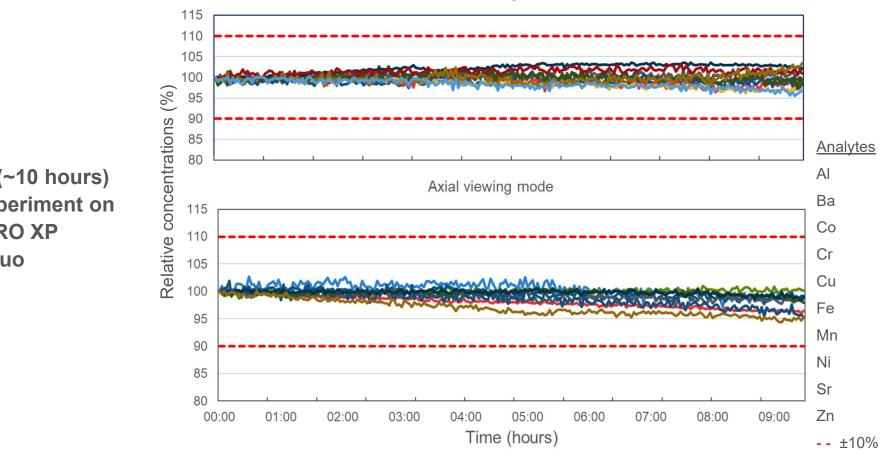
Long-term (18 hours) experiment on the iCAP PRO XP ICP OES, Radial only instrument



---±10%

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Results – Long-Term Stability Experiments on the Duo Instrument



Radial viewing mode

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Long-term (~10 hours) stability experiment on the iCAP PRO XP ICP- OES Duo

- Increased operation time with minimal downtime
- Reduced maintenance requirements and improved robustness cost effective.
 No ICP torch failure, no cleaning of torch and cones necessary
- No problem analyzing high matrix samples like 25% NaCl solutions, i.e., seawater-like samples over uninterrupted long time periods (at least 10 hours)
 - Radial only ✓
 - Duo 🗸
- Dilution steps for high matrix samples eliminated vertical ceramic torch and sheath gas adaptor easy to install
- Better detection limits and enhanced sensitivity in Axial mode



Conclusion – Benefits for Environmental Analysis

