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S C I E N T I F I C

# ICP-OES - Solutions for complex matrices

16:th Nordic User Meeting on AAS, ICP-OES and ICP-MS

*Presented by*  
**Mikael Axelsson ThermoFisher Scientific Nordic**

The world leader in serving science



Complex matrices



ICP-OES



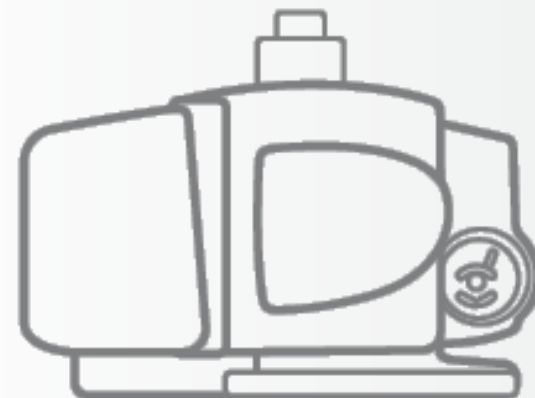
Application examples



Summary

# An ideal world

- An ideal sample would have little or no matrix
  - Matrix causes differences in sample transportation
  - High TDS causes blockages
  - Volatility of organics
  - Different matrix components will cause different interferences
- Ideally matrix match all samples and standards
- Instrument set up important



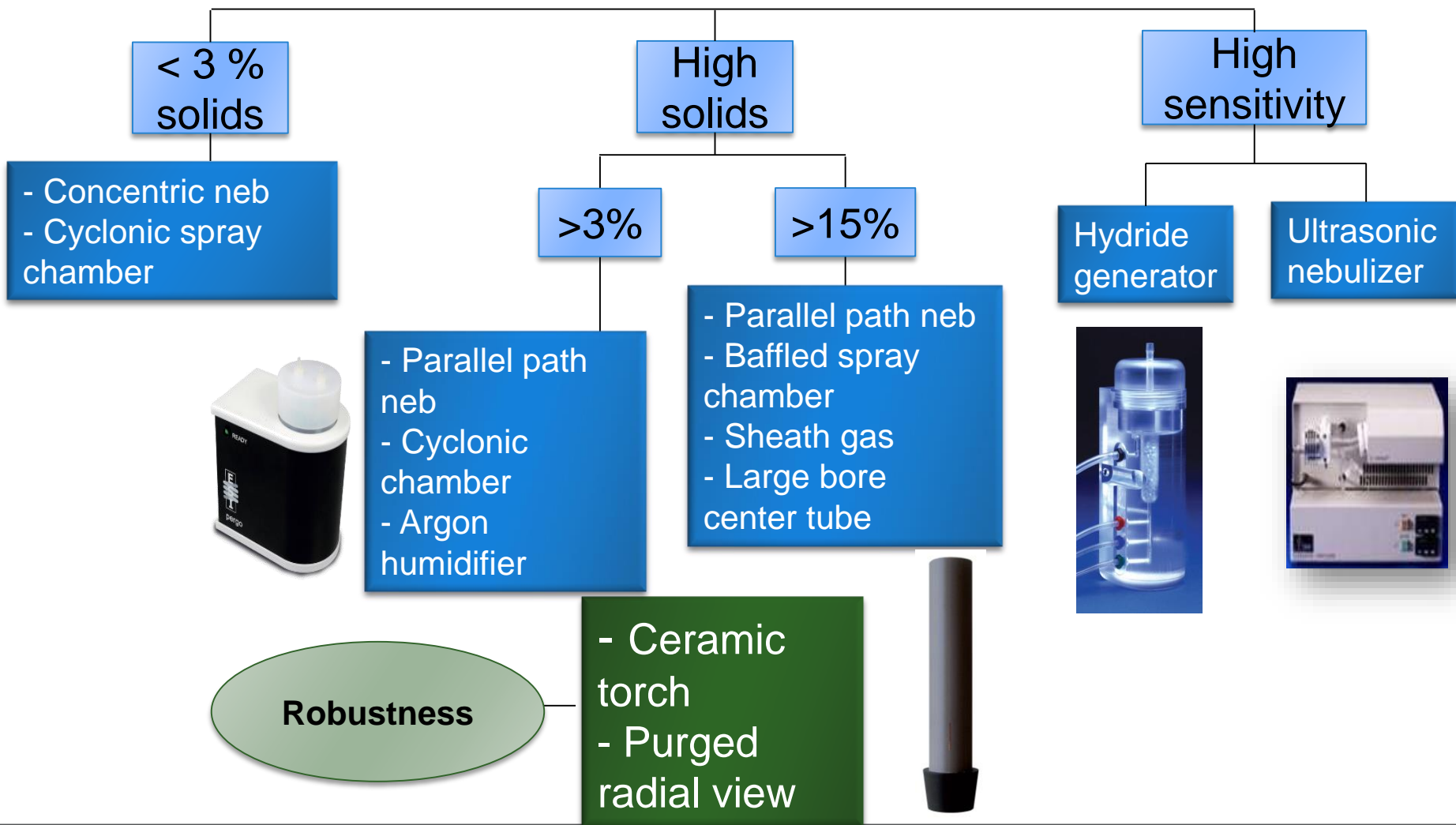
# Thermo Scientific™ iCAP™ 7000 Plus Series ICP-OES

- *iCAP 7200 ICP-OES* - Entry level Duo ICP, cost-effective analysis for low sample thru-put requirements
- *iCAP 7400 ICP-OES* – Duo or Radial ICP for routine analysis requirements and mid-range sample thru-put. Duo or Radial
- *iCAP 7600 ICP-OES* – Duo or Radial ICP for highest productivity and maximum sample thru-put with advanced, flexible accessory support, such as laser ablation

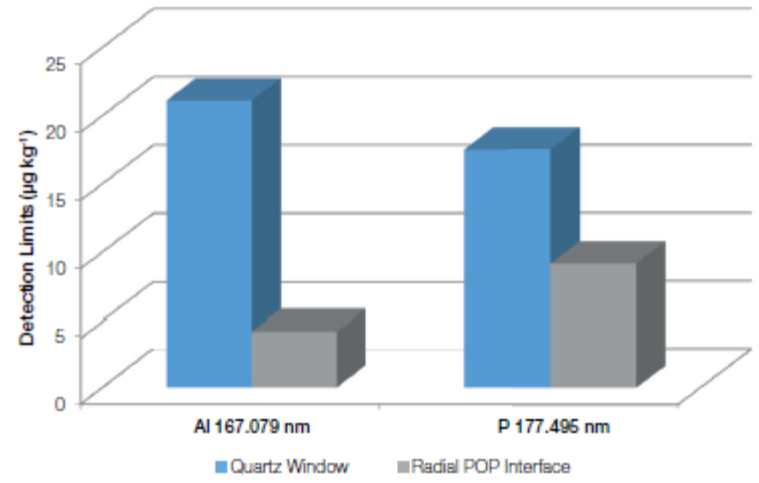
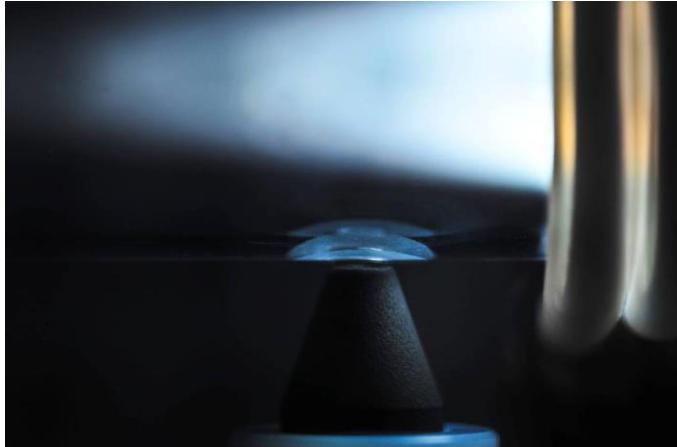
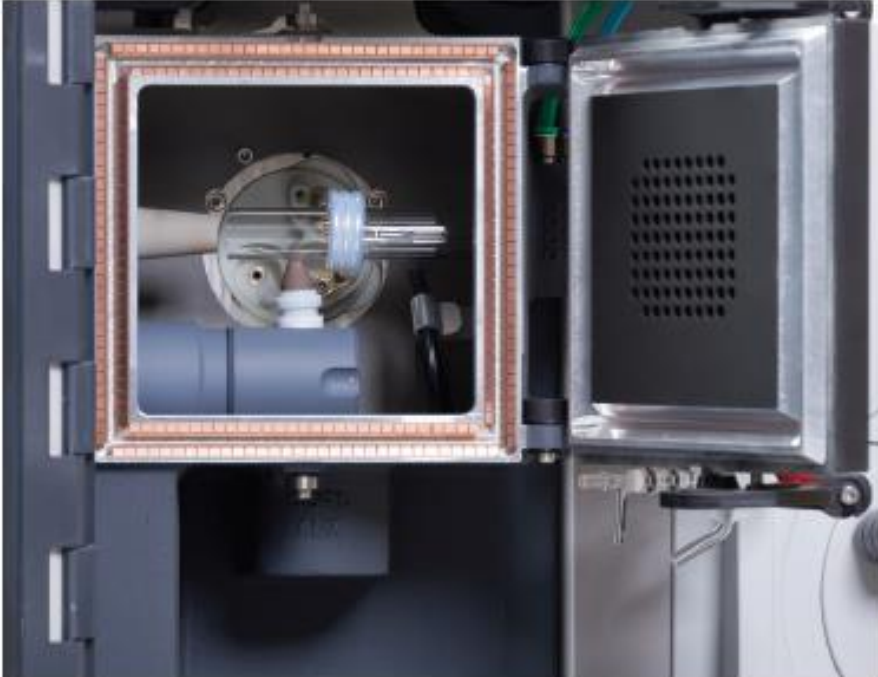


# Selection of Sample Introduction

## Aqueous

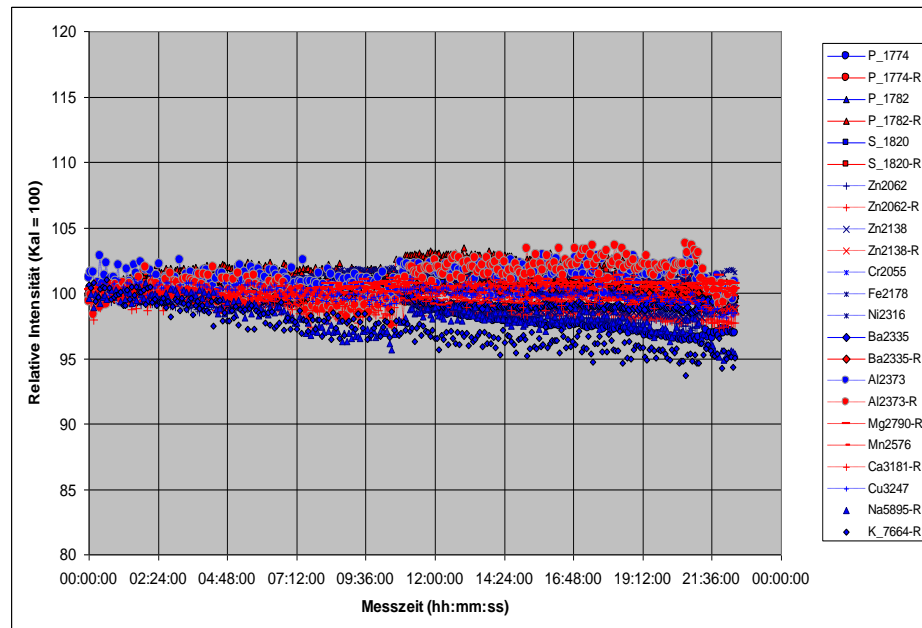
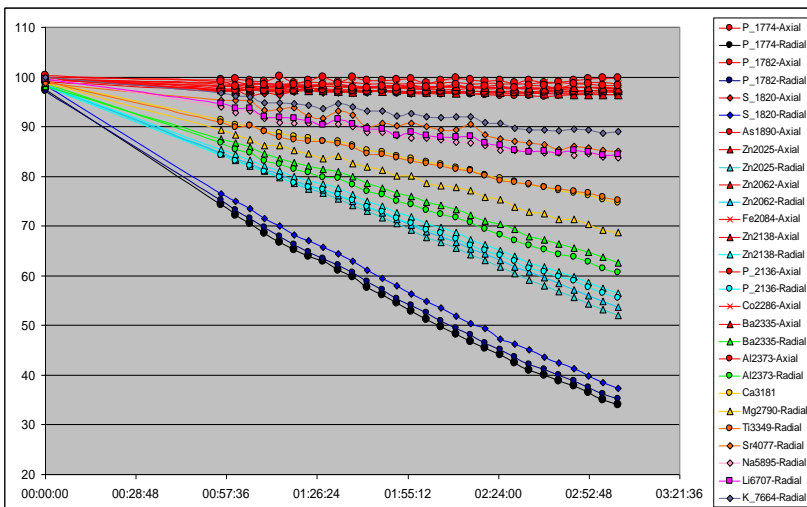


# iCAP Duo - purged optical path



# Metal concentrates

## Aqua regia 10g/L metal concentrate



Axial (red) and radial (blue) performance shows no difference

Same solution with standard quartz radial view tube, axial stable and radial drift over time

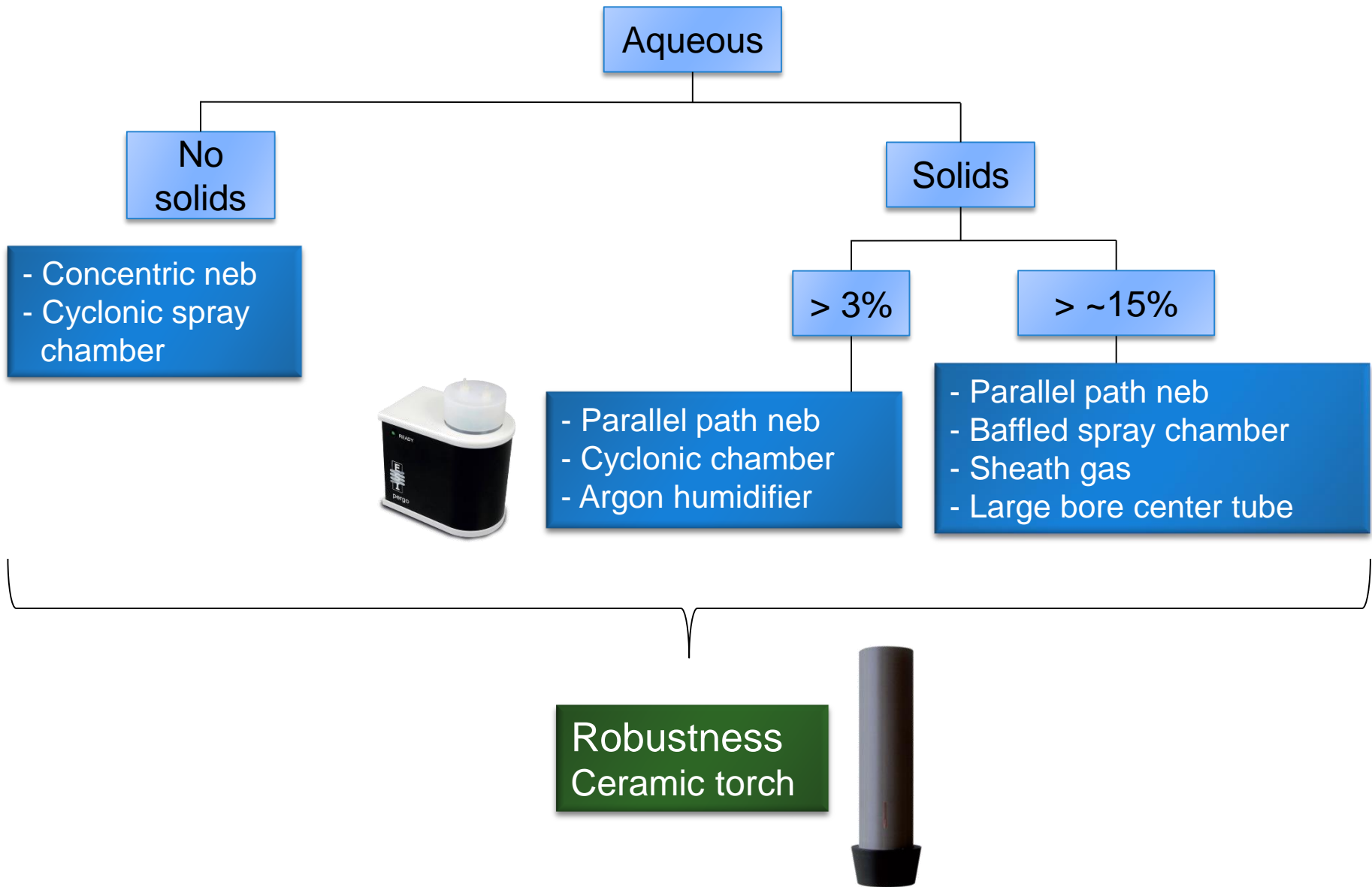
# Metal concentrates

300 measured samples – normalised to 100% and monitored

Line	Observation	Avg	RSD	min	max	$\Delta$
	ax/rad	%	%	%	%	%
P_1774	axial	98,6	1,2	96,1	100,0	3,9
P_1774-2	radial	100,5	0,6	98,4	101,5	3,1
P_1782	axial	100,5	0,6	98,7	101,4	2,7
P_1782-2	radial	102,0	0,7	100,0	103,5	3,5
S_1820	axial	100,6	0,4	99,3	101,4	2,2
S_1820-2	radial	100,8	0,5	99,2	102,0	2,8
As1890	axial	99,6	0,6	97,7	101,2	3,5
Zn2025	axial	96,5	1,9	93,1	100,1	7,0
Zn2025-2	radial	96,8	1,7	93,9	99,9	6,0
Cr2055	axial	100,1	0,4	99,0	101,0	2,0
Zn2062	axial	99,0	0,9	97,2	100,9	3,7
Zn2062-2	radial	99,0	0,6	97,4	100,1	2,7
Fe2084	axial	98,9	1,0	97,1	100,2	3,1
P_2136	axial	100,1	0,4	99,3	101,1	1,8
P_2136-2	radial	100,2	0,3	99,3	100,9	1,6
Zn2138	axial	99,2	0,5	98,1	100,2	2,1
Zn2138-2	radial	99,9	0,3	98,5	100,5	2,1
Fe2178	axial	100,1	0,3	98,9	100,7	1,8
Pb2203	axial	100,9	0,6	99,8	102,2	2,4
Ni2216	axial	101,2	0,8	99,7	102,6	2,9
Co2286	axial	99,9	0,5	99,2	101,2	2,0
Ni2316	axial	101,0	0,5	99,9	102,0	2,1
Ba2335	axial	99,5	0,5	98,5	100,9	2,4
Ba2335-2	radial	100,2	0,3	99,3	101,1	1,8



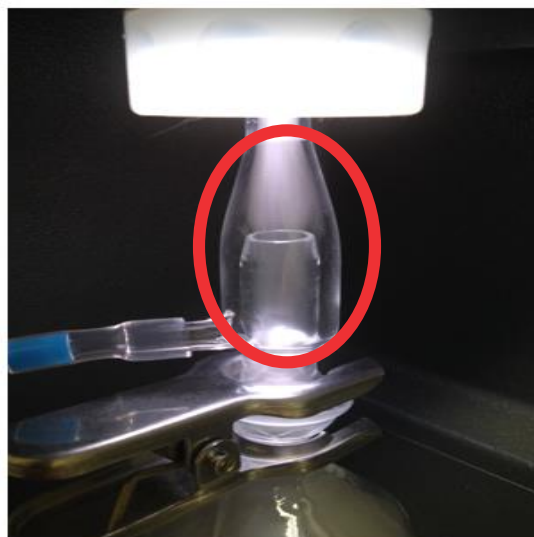
# Argon Humidifier or Sheath Gas



# Sheath Gas for 7400 and 7600 Radial

- Sheath Gas improves the long term stability of instrument when running high salt applications
- Isolates the sample aerosol and prevents build up of salt crystals in the torch / Injector
- Allows users to run for longer

Sheath Gas off

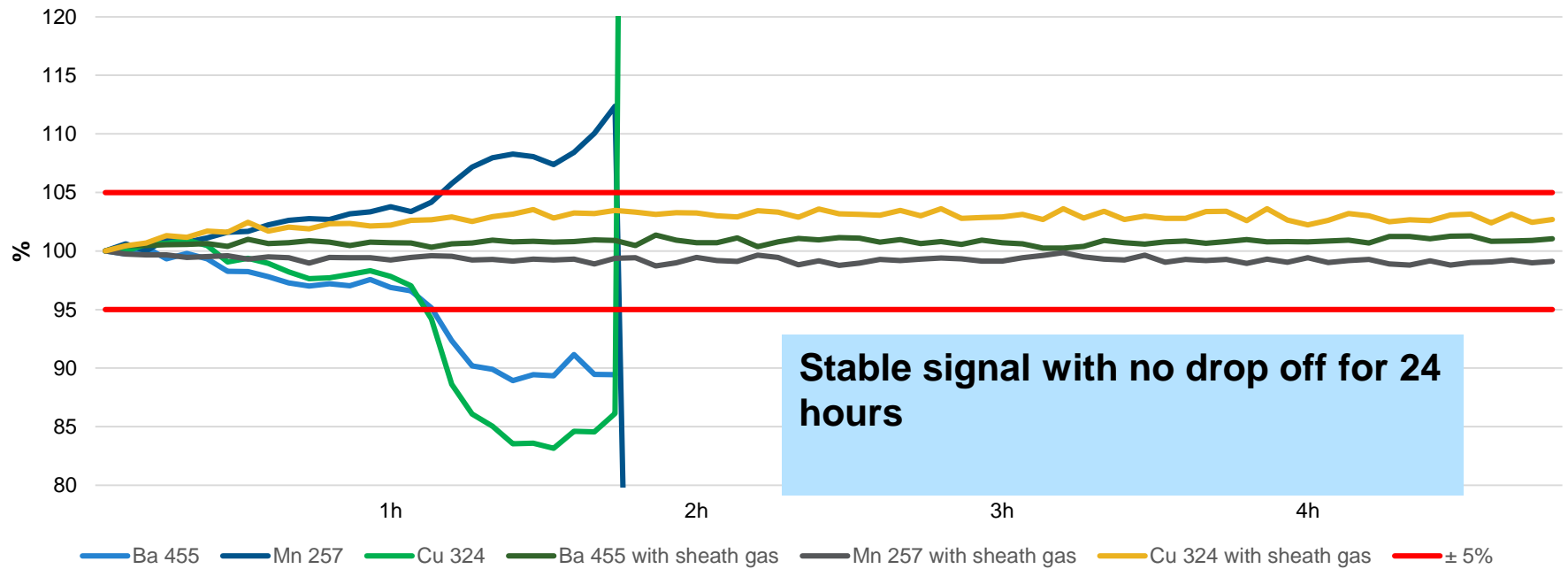


Sheath Gas on



# Stability data comparison with and without Sheath Gas

## Stability 25% NaCl with and without Sheath Gas



# Combining Sheath gas and D-Torch

- Combine both the D-Torch and the sheath gas for the ultimate matrix tolerance
- No special parameters
  - Coolant gas just 12 l/min
- No extended wash periods
  - Just 30 seconds
- Additional gas flow minimal
  - Just 100ml/min argon

Parameter	Setting
Pump Tubing (Standard Pump)	Sample Tygon® orange/white Drain Tygon® white/white
Spray Chamber	Baffled cyclonic
Nebulizer	Burgener Mira Mist
Center Tube	2.0 mm (ceramic)
Torch	Ceramic D-Torch
Pump Speed	50 rpm
Flush Pump Speed	100 rpm
Pump Stabilization Time	10 s
Wash Time	30 s
Nebulizer Gas Flow	0.55 L·min <sup>-1</sup>
Auxiliary Gas Flow	0.5 L·min <sup>-1</sup>
Coolant Gas Flow	12 L·min <sup>-1</sup>
Additional Gas Flow	0.1 L·min <sup>-1</sup>
RF Power	1400 W
Radial Viewing Height	10 mm
Exposure Time	UV 15 s, Vis 15 s

# Excellent recoveries and performance

- Calibration standards prepared in 25% NaCl
- 25% NaCl samples analyzed for an extended period
- Excellent spike recoveries

Element and wavelength (nm)	R <sup>2</sup>	Measured spike concentration (µg·L <sup>-1</sup> )	Recovery (%)	MDL (µg·L <sup>-1</sup> )
Al 167.079	0.9997	497.2	97.2	4.1
Ba 455.403	1.0000	499.9	99.9	0.9
Co 228.616	0.9999	500.6	100.6	10.6
Cr 205.560	0.9999	500.5	100.5	4.5
Cu 324.754	0.9999	501.7	101.7	13.0
Fe 238.204	0.9995	503.2	103.6	8.8
Mg 279.553	0.9999	501.0	101.0	0.5
Mn 257.610	0.9999	501.1	101.1	1.7
Ni 221.647	0.9999	500.8	100.8	8.0
Sr 216.596	0.9999	501.3	101.3	10.2
Zn 202.548	1.0000	499.9	99.9	2.0

# Stability and Robustness

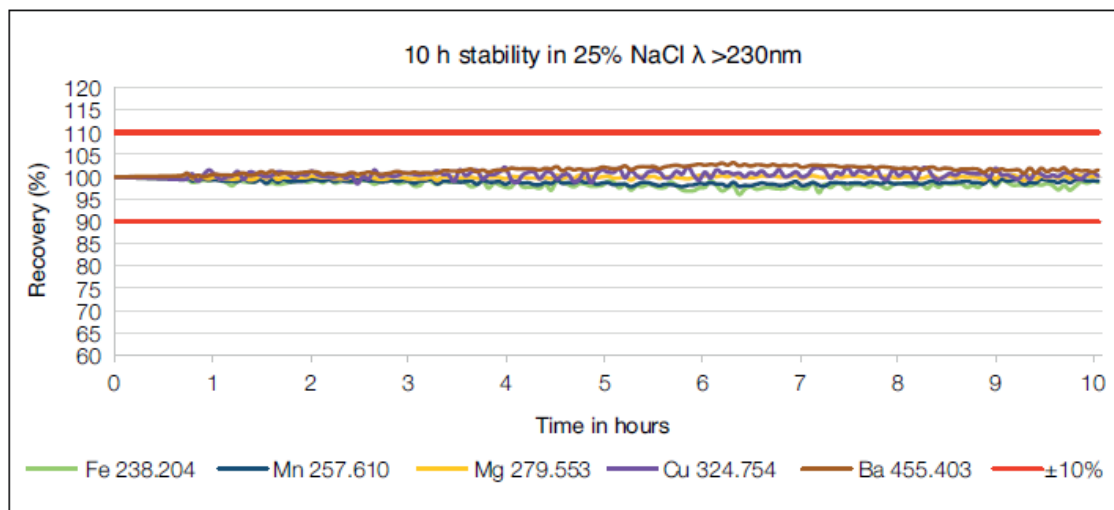


Figure 4. Results obtained for the 10 hour stability analysis (wavelengths > 230 nm).

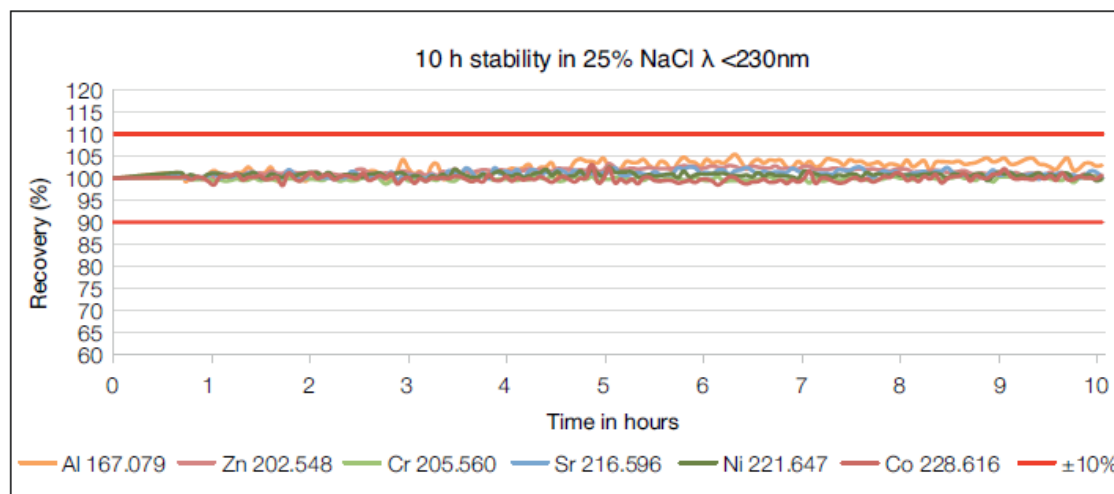



Figure 5. Results obtained for the 10 hour stability analysis (wavelengths < 230 nm).


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PRODUCT SPOTLIGHT



## Thermo Scientific iCAP 7000 Plus Series ICP-OES

Performance and stability for high concentration salt solutions using a sheath gas




The sheath gas adaptor for the Thermo Scientific™ iCAP™ 7000 Plus Series ICP-OES allows samples with a high concentration of total dissolved solids to be analyzed over extended periods of time, reducing user maintenance and increasing long term stability in these challenging matrices.

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APPLICATION NOTE 43444



## High matrix tolerance of the Thermo Scientific iCAP 7000 Plus Series ICP-OES with the radial Ceramic D-Torch and the sheath gas adaptor

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**Keywords**  
Ceramic D-Torch, high matrix, NaCl, robustness, salt, sheath gas adaptor

**Introduction**  
The sample introduction system of an ICP-OES has a large influence on the analytical performance of the spectrometer and is the main area of the hardware that users interface with. One key component of this system is the ICP torch. The ICP torch is a relatively high cost consumable item, which can require regular maintenance and replacement when performing more demanding applications. Currently, the majority of ICP torches are made from quartz, which is a crystalline form of silicon dioxide (silica). When a quartz ICP torch is heated (by the plasma) it can undergo a process known as devitrification (which means becoming less glasslike). The process of devitrification can decrease the expected lifetime of the ICP torch and is commonly seen when samples are analyzed that contain high concentrations (greater than 1000 mg/L) of group I or group II elements (Figure 1).

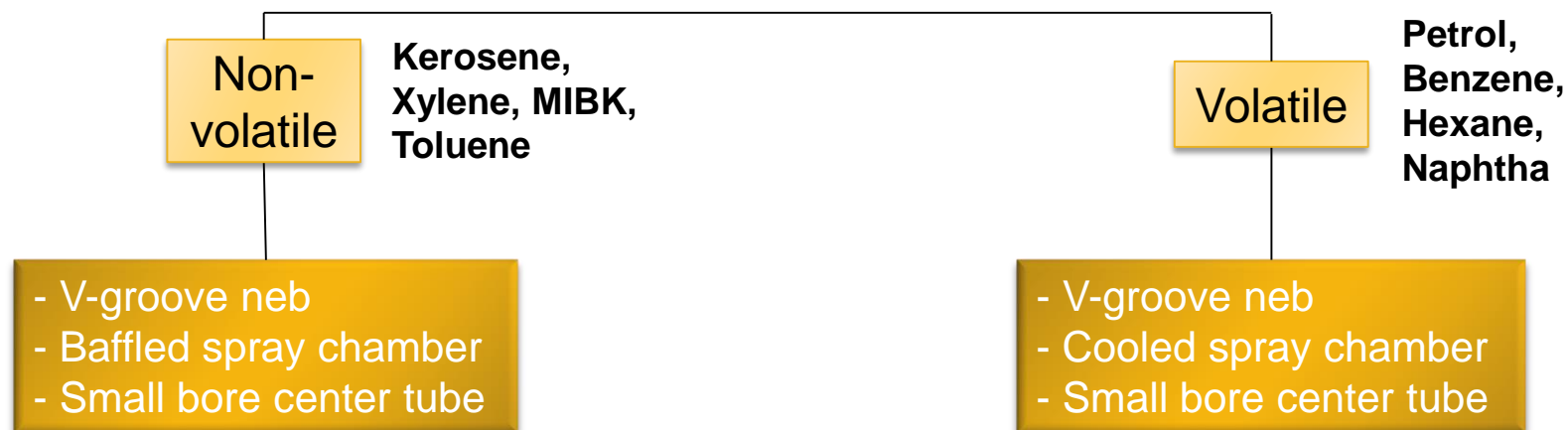
**Goal**  
This application note describes how routine analysis of high matrix samples like 25% NaCl can be performed with the Thermo Scientific iCAP 7400 ICP-OES Radial fitted with the additional gas accessory in combination with the Ceramic D-Torch and the sheath gas adaptor.

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- Greater robustness for extend periods of time
- Minimal sample preparation – less or no dilution of samples

# Selection of Sample Introduction

## Organics

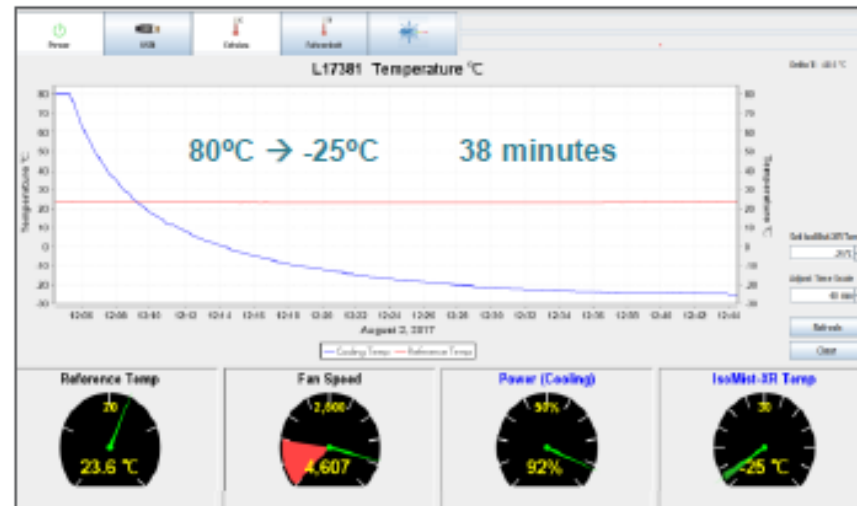
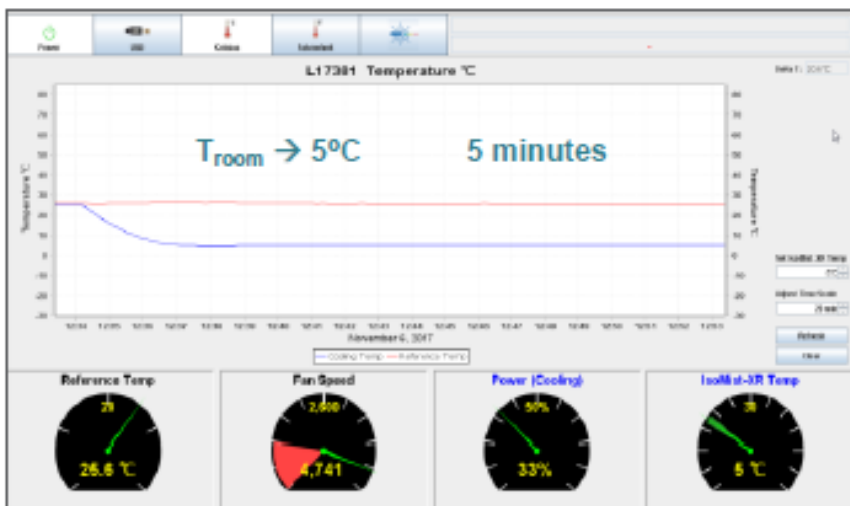
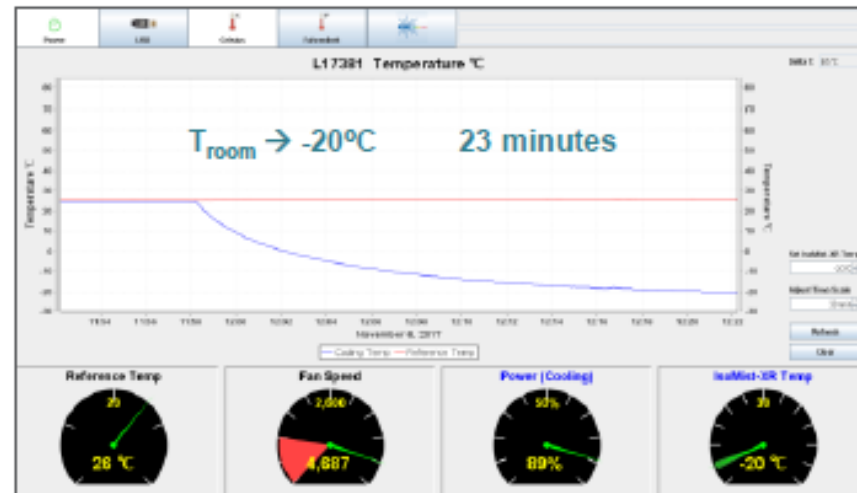


Chemical resistant pump tubing



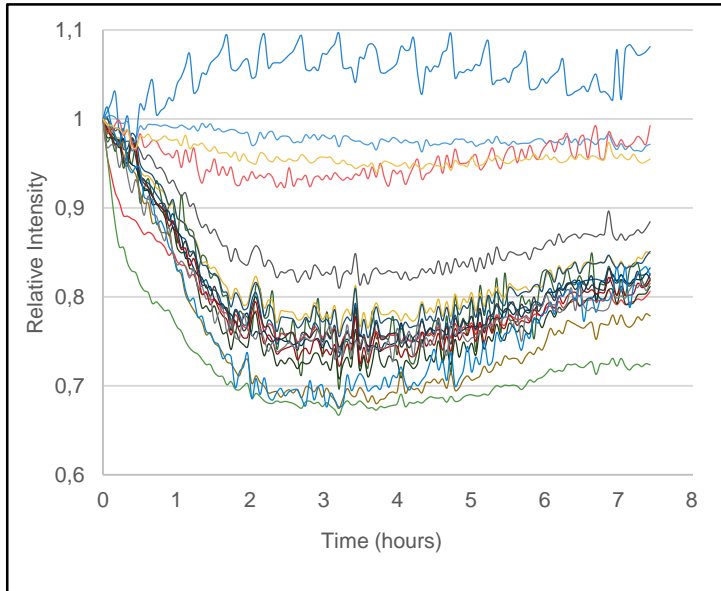


# Isomist XR

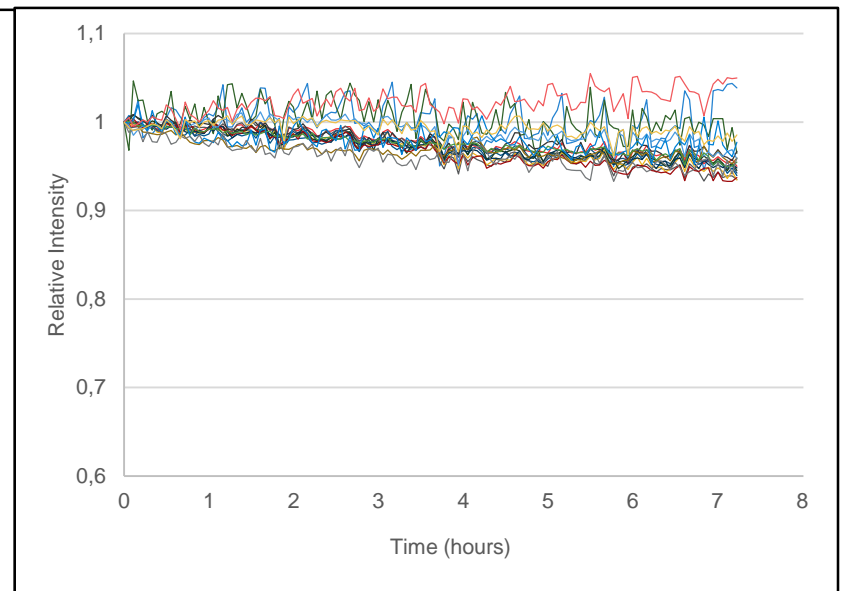


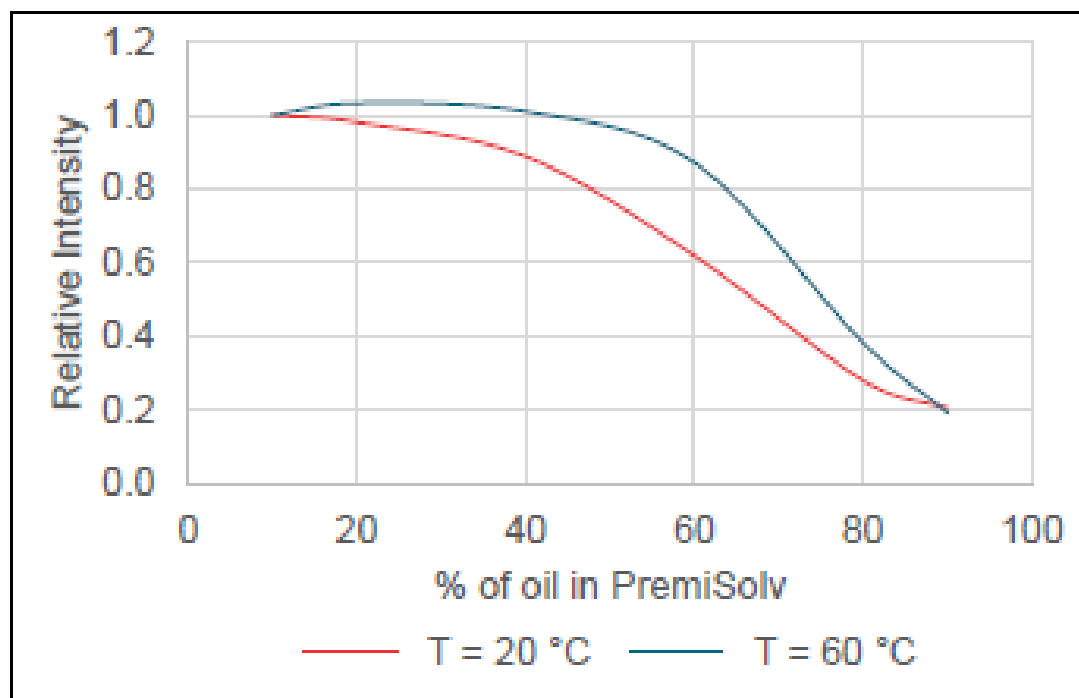
# Analysis of Naphtha with IsoMist XR

- IsoMist XR
  - Temperature range -25 °C to +80 °C
  - 8h stability measurement without temperature control



- 8h stability measurement controlled at -20°C

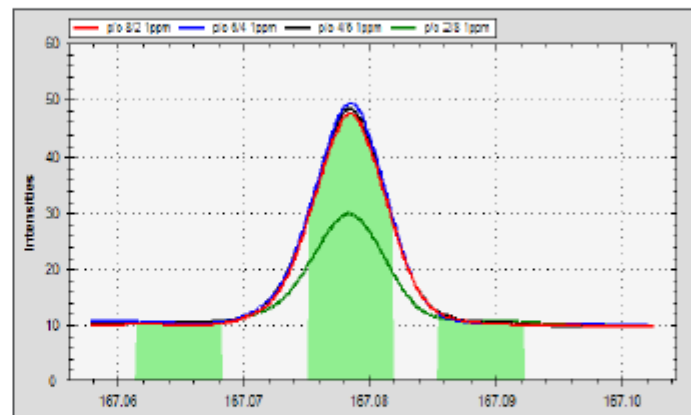
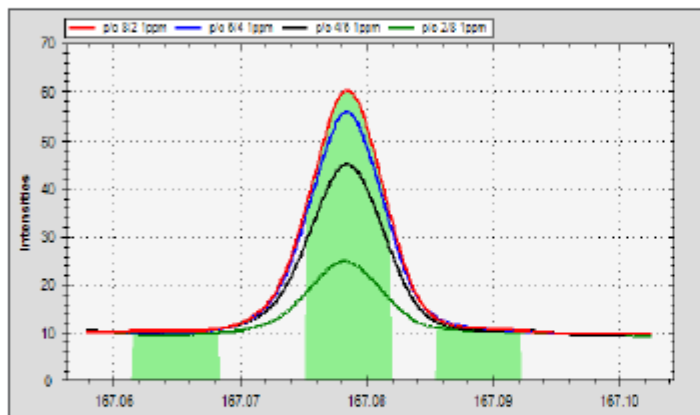




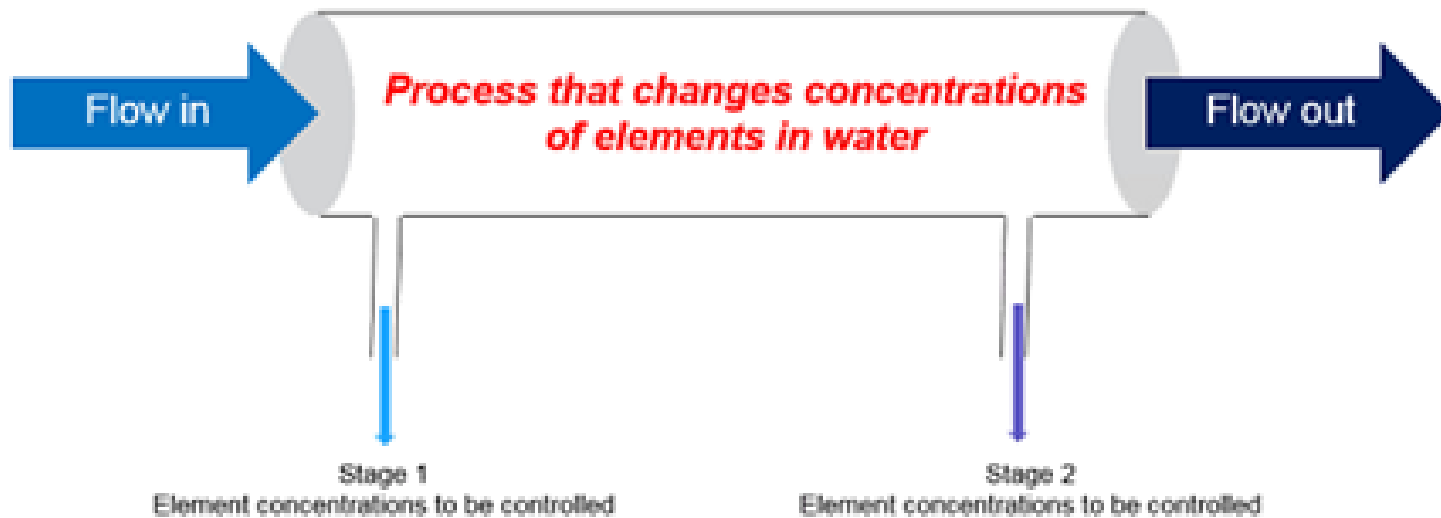
By increasing the temperature it is possible to reduce the viscosity of samples and therefore to enhance signals in ICP-OES measurements (Figure 7).

# Content of oil in sample

Average relative intensities for 1 mg·kg<sup>-1</sup> aluminum at wavelength 167.079 nm, with pre-set temperatures at 20°C (left) and 60°C (right), depending on the content of oil in PremiSolv.

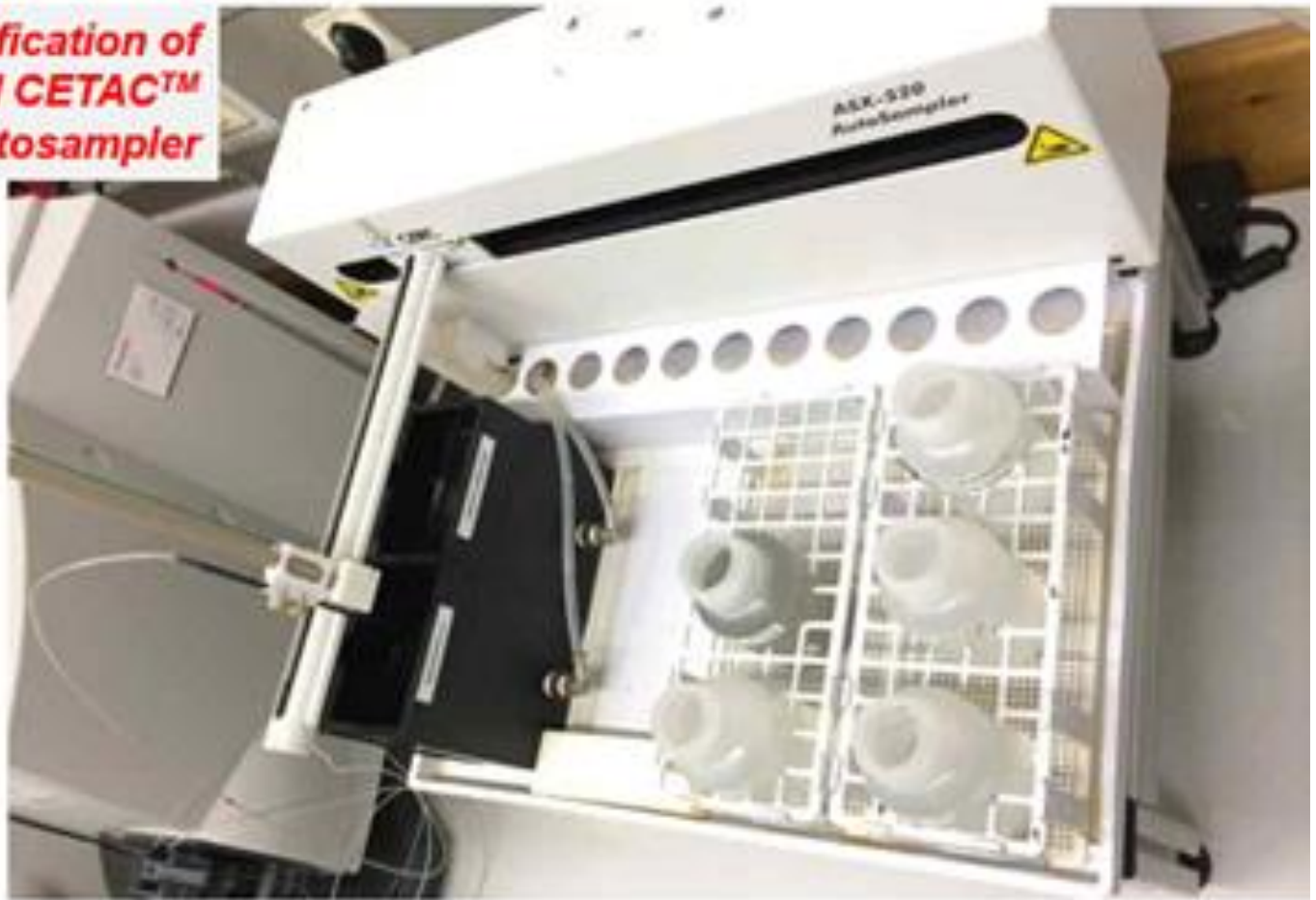


*How a process can look like*



# Sewage process monitoring – 2 stages

**modification of  
a standard CETAC™  
ASX-520 Autosampler**



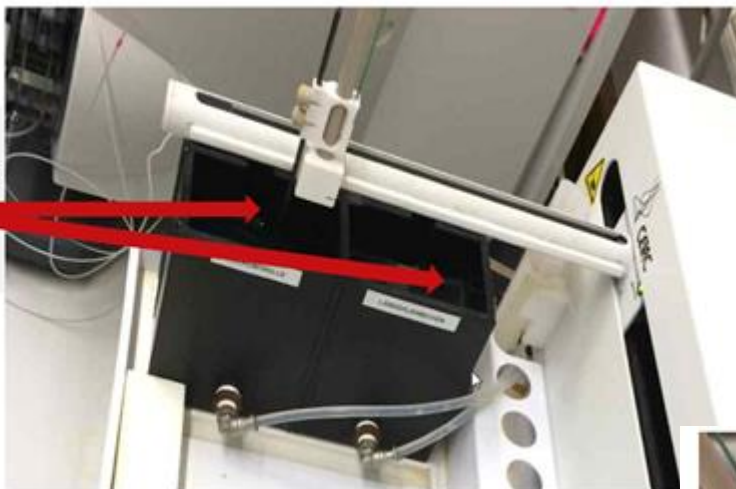
# Continuous control of elements in water online

1 blank,  
2 calibration standards,  
1 QC sample and  
1 optional sample space for  
any emergency case

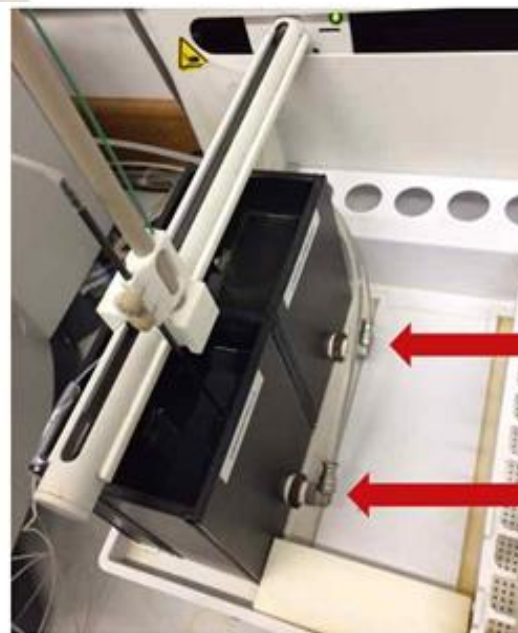


# Feed sample analysis

custom-made racks with 2 overflow basins for water, continuously collected at 2 different stages



- Algae growth
- Internal standard
- Pump tube wear
- Particles in sample
- Rinse station flow





# Qtegra Labbook to run samples online for over 24 hours

Use long Exposure Time + long Wash and Uptake Time + **numbers of readings** to get required work time

Sample list estimated runtime: 1 days 3 hours 49 minutes 20 seconds

	Label	Repeats	Full Frames	Comments	Est. User	Sample Type	Standard
1	Blank	3		<Comment>	✓	STD	Blank
	Std-Abrasser 1	3		<Comment>	✓	STD	Std-Abrasser 1
	Std-Abrasser 2	3		<Comment>	✓	STD	Std-Abrasser 2
	Kontrollstandard	3		<Comment>	✓	QC	QC Std-Abrasser 3
	Kanal 2	5		<Comment>	✓	UNKNOWN	
	Kanal 1	5		<Comment>	✓	UNKNOWN	
	Kanal 2	5		<Comment>	✓	UNKNOWN	
	Kanal 1	5		<Comment>	✓	UNKNOWN	
	Kanal 2	5		<Comment>	✓	UNKNOWN	
	Kanal 1	5		<Comment>	✓	UNKNOWN	
	Kontrollstandard	3		<Comment>	✓	QC	QC Std-Abrasser 3
	Kanal 1	5		<Comment>	✓	UNKNOWN	
	Blank	3		<Comment>	✓	STD	Blank
	Std-Abrasser 1	3		<Comment>	✓	STD	Std-Abrasser 1
	Std-Abrasser 2	3		<Comment>	✓	STD	Std-Abrasser 2
	Kontrollstandard	3		<Comment>	✓	QC	QC Std-Abrasser 3
	Kanal 1	5		<Comment>	✓	UNKNOWN	
	Kanal 2	5		<Comment>	✓	UNKNOWN	
	Kanal 1	5		<Comment>	✓	UNKNOWN	
	Kontrollstandard	3		<Comment>	✓	QC	QC Std-Abrasser 3

**386 samples**

**in 1 days 3 hours 49 minutes**

- It is possible to analyse challenging sample types by ICP-OES
- Careful optimisation of the method parameters are required to get the best possible performance
- Selection of the appropriate sample introduction components is essential
- ICP-OES can be used for online sample analysis for real time trace element measurements.

