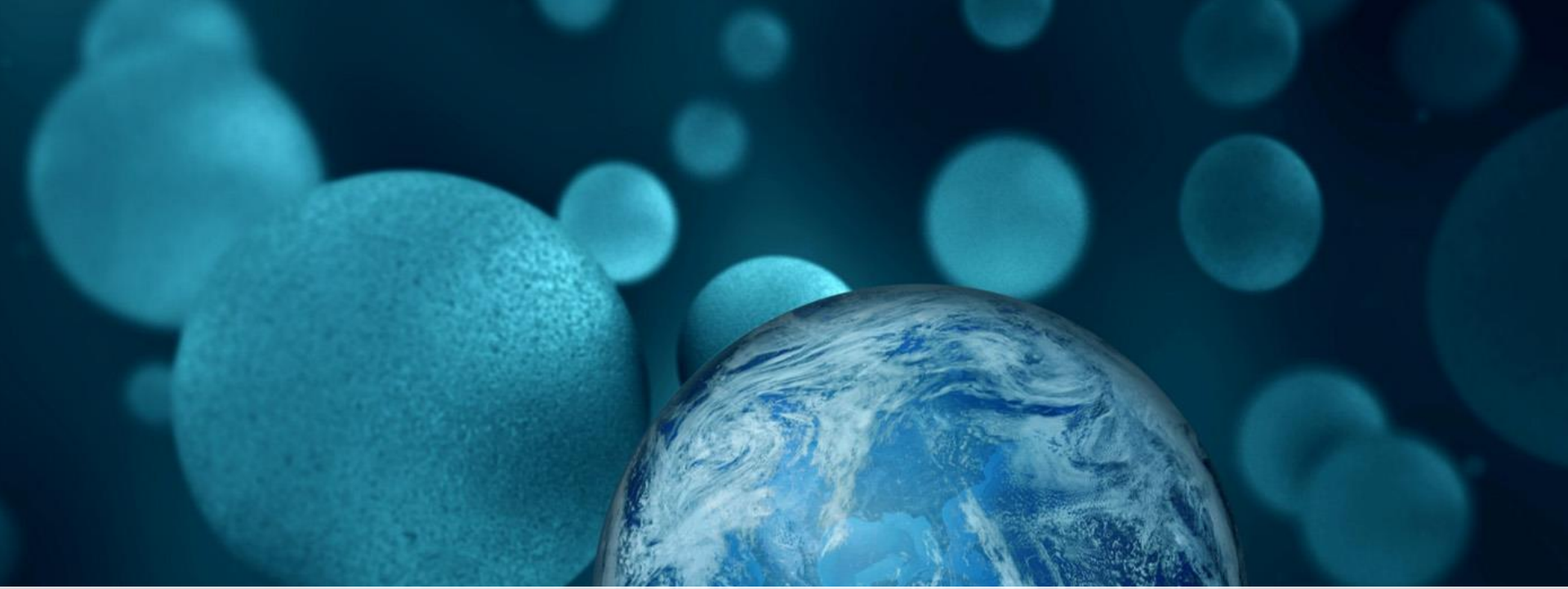


ThermoFisher SCIENTIFIC

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

Overview

Complex matrices

ICP-OES

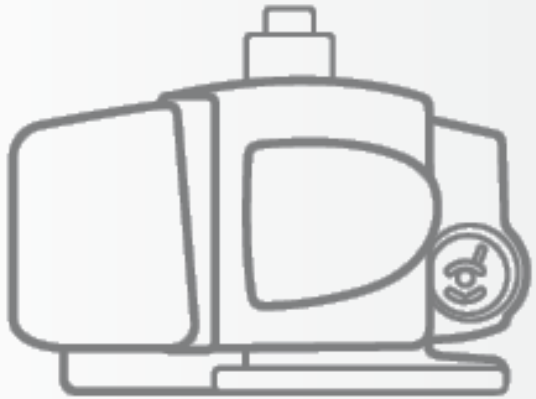
Application examples

Summary

ThermoFisher SCIENTIFIC

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

ThermoFisher SCIENTIFIC

Selection of Sample Introduction

Aqueous

< 3 % solids

- Concentric neb
- Cyclonic spray chamber

High solids

>3%

- Parallel path neb
- Cyclonic chamber
- Argon humidifier

>15%

- Parallel path neb
- Baffled spray chamber
- Sheath gas
- Large bore center tube

Robustness

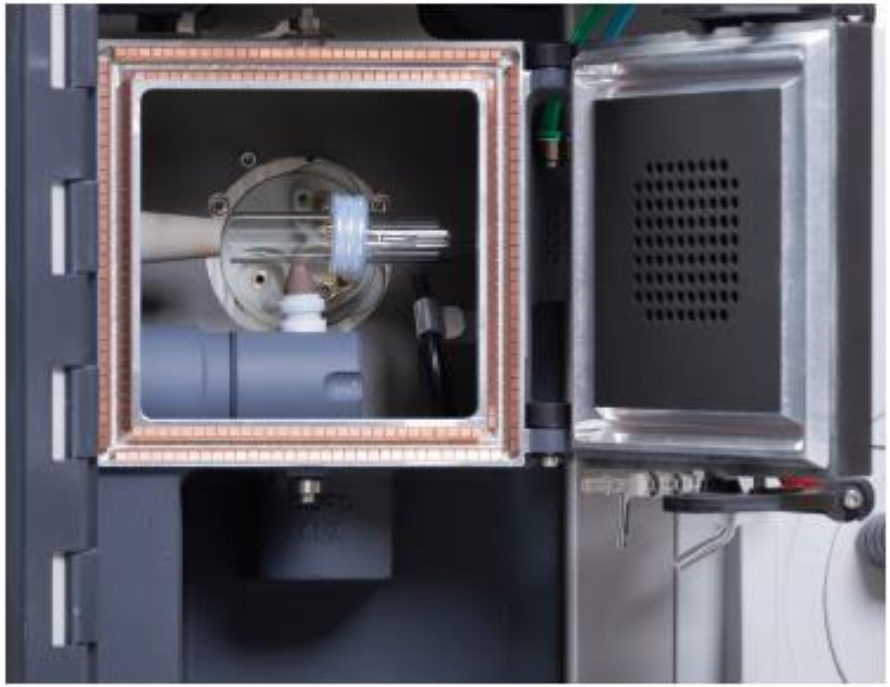
- Ceramic torch
- Purged radial view

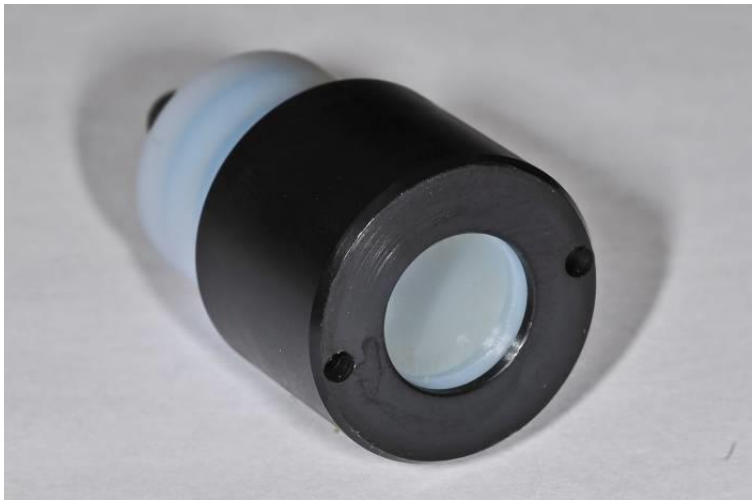
High sensitivity

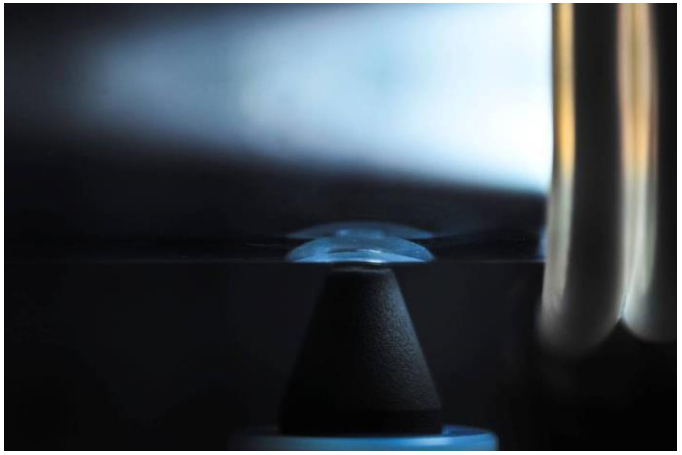
Hydride generator

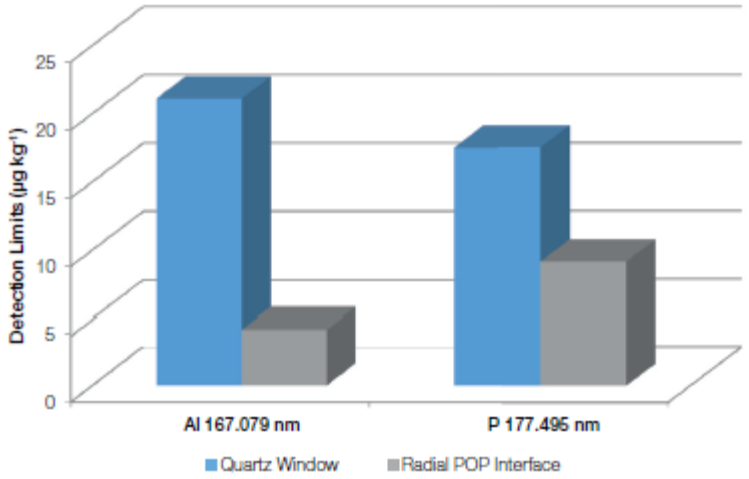
Ultrasonic nebulizer

iCAP Duo - purged optical path





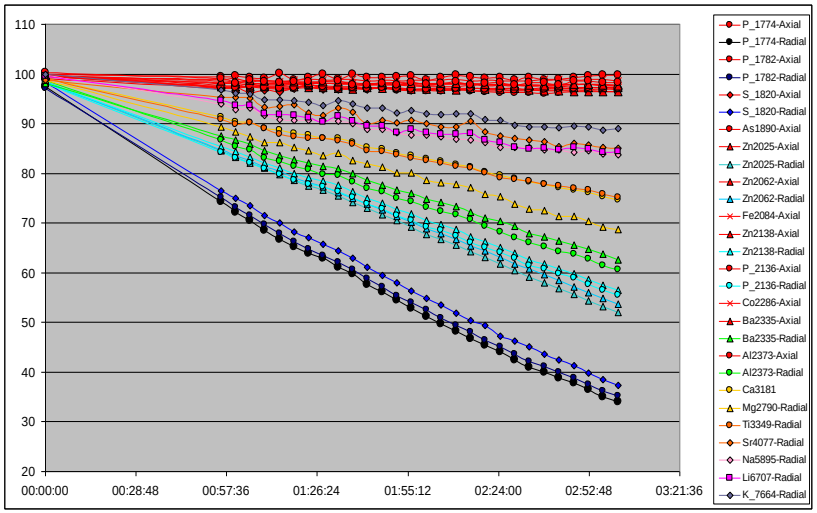


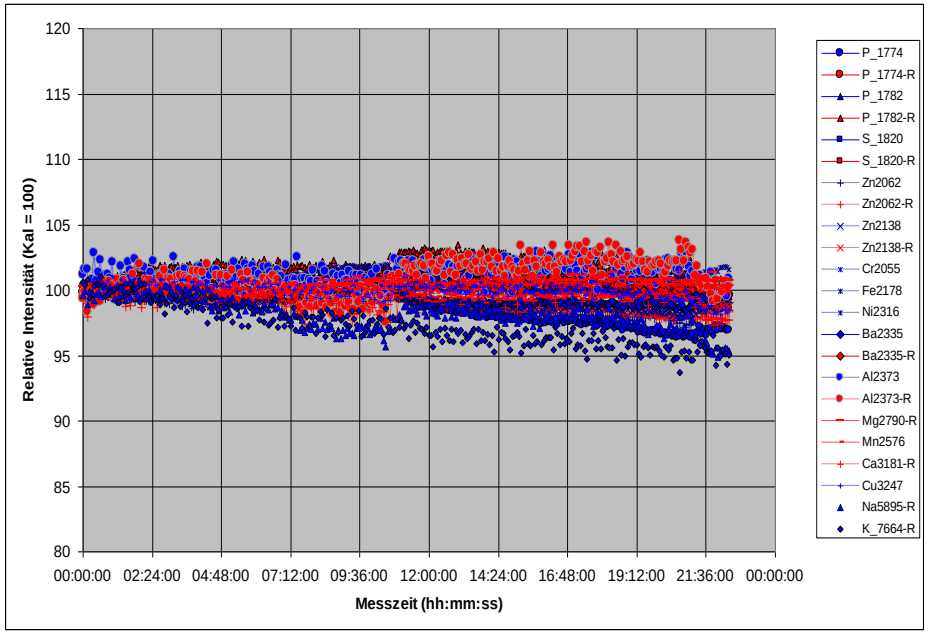


ThermoFisher SCIENTIFIC

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	Δ
	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

Aqueous

No solids

- Concentric neb
- Cyclonic spray chamber

Solids

> 3%

- Parallel path neb
- Cyclonic chamber
- Argon humidifier

> ~15%

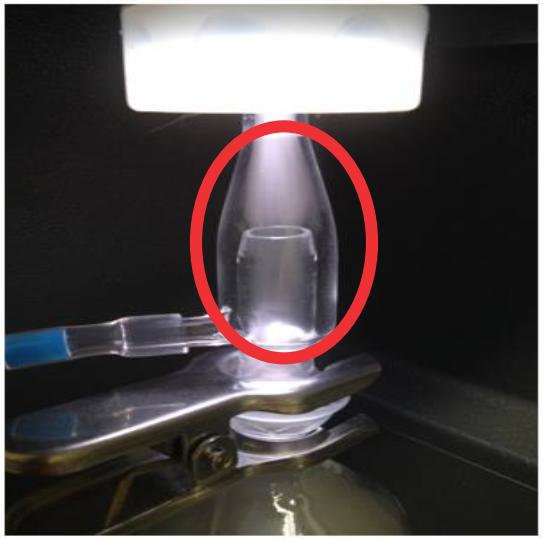
- Parallel path neb
- Baffled spray chamber
- Sheath gas
- Large bore center tube

Robustness
Ceramic torch

Sheath Gas for 7400 and 7600 Radial

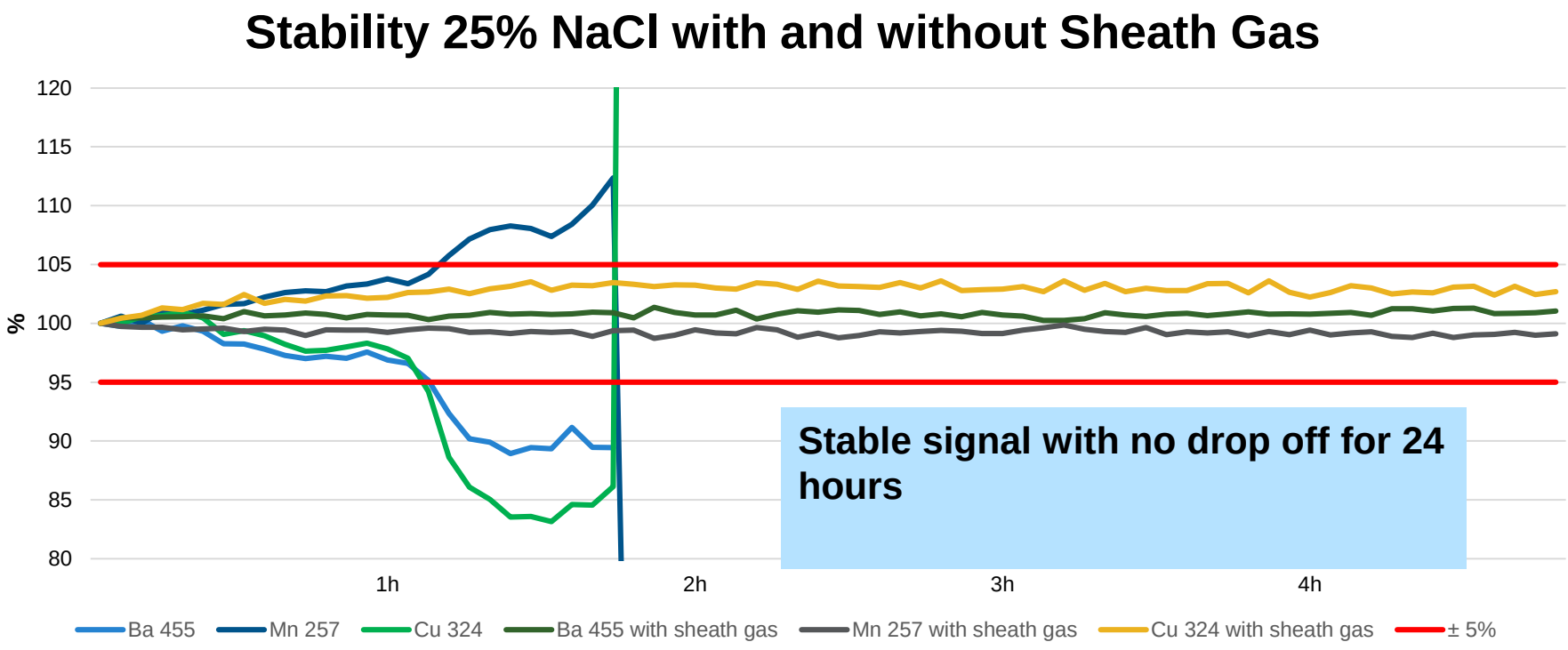
- Sheath Gas improves the long term stability of instrument when running high salt applications
- Isolates the sample aerosol and prevents build up of salt crystals in the torch / Injector
- Allows users to run for longer

Sheath Gas off



Sheath Gas on

Stability data comparison with and without Sheath Gas



ThermoFisher SCIENTIFIC

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 $L \cdot min^{-1}$
Auxiliary Gas Flow	0.5 $L \cdot min^{-1}$
Coolant Gas Flow	12 $L \cdot min^{-1}$
Additional Gas Flow	0.1 $L \cdot min^{-1}$
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^2	Measured spike concentration ($\mu g \cdot L^{-1}$)	Recovery (%)	MDL ($\mu g \cdot L^{-1}$)
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

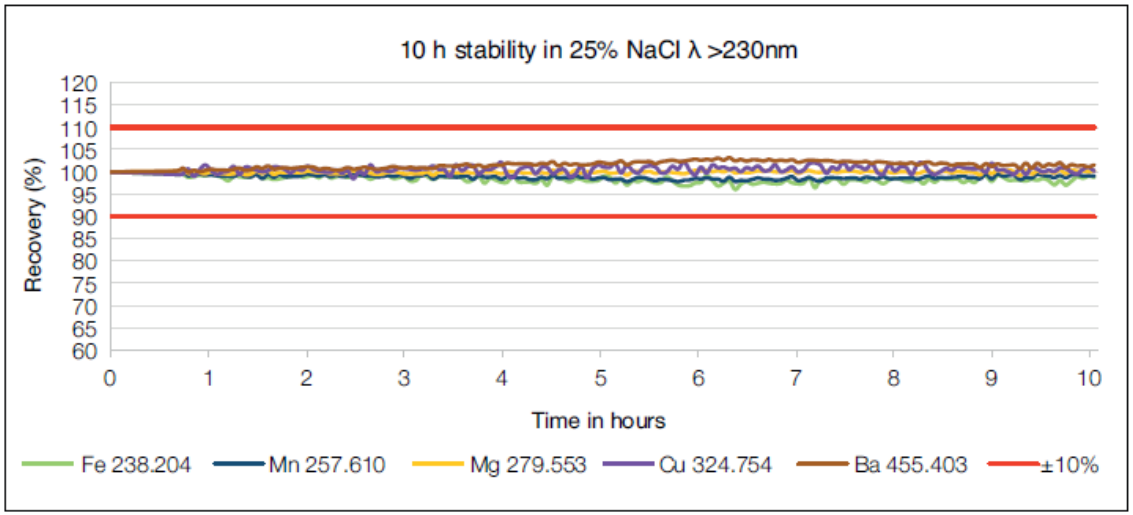


Figure 4. Results obtained for the 10 hour stability analysis (wavelengths > 230 nm).

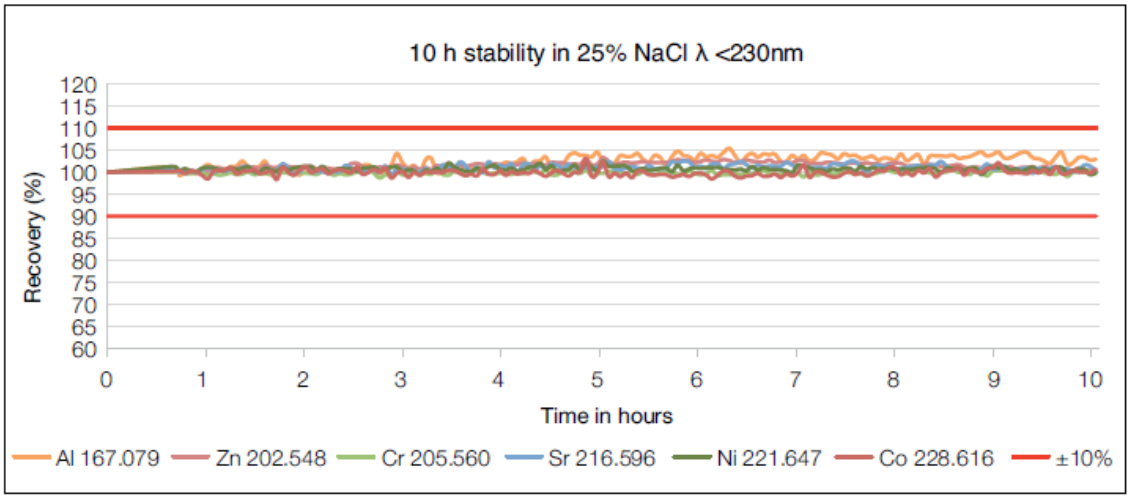
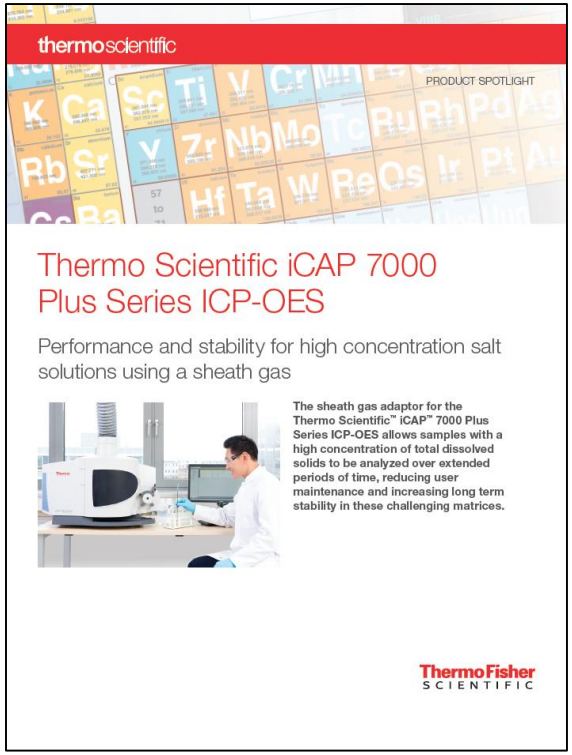
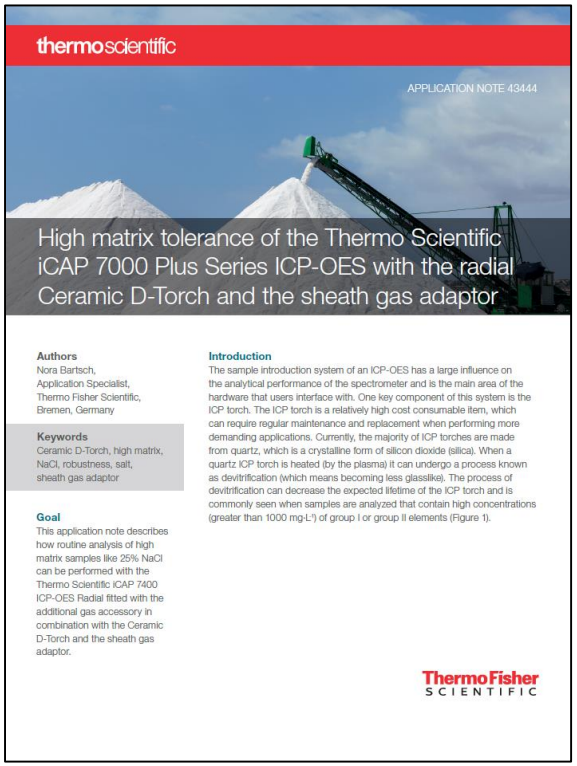


Figure 5. Results obtained for the 10 hour stability analysis (wavelengths < 230 nm).

ThermoFisher SCIENTIFIC

Application notes





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

ThermoFisher SCIENTIFIC

Selection of Sample Introduction

Organics

Non-volatile

Kerosene, Xylene, MIBK, Toluene

- V-groove neb
- Baffled spray chamber
- Small bore center tube

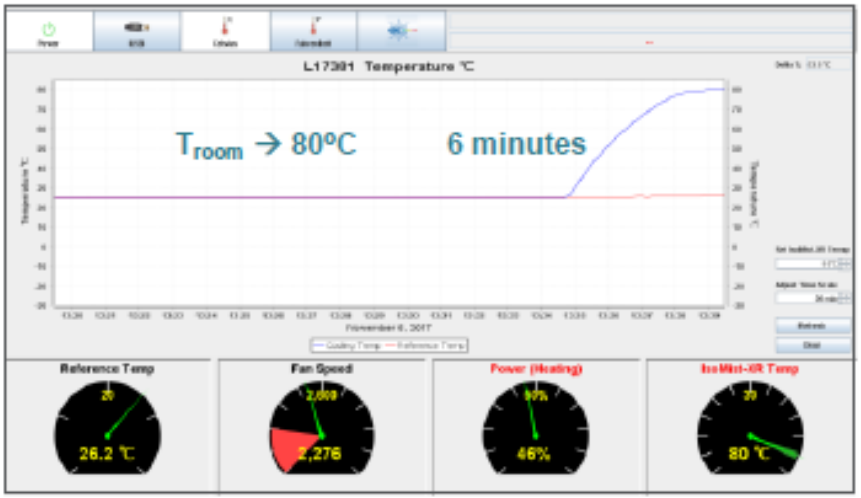
Volatile

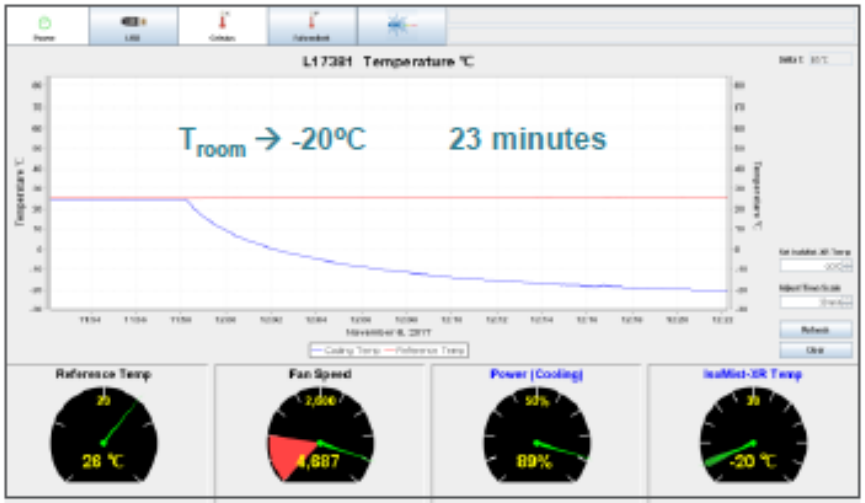
Petrol, Benzene, Hexane, Naphtha

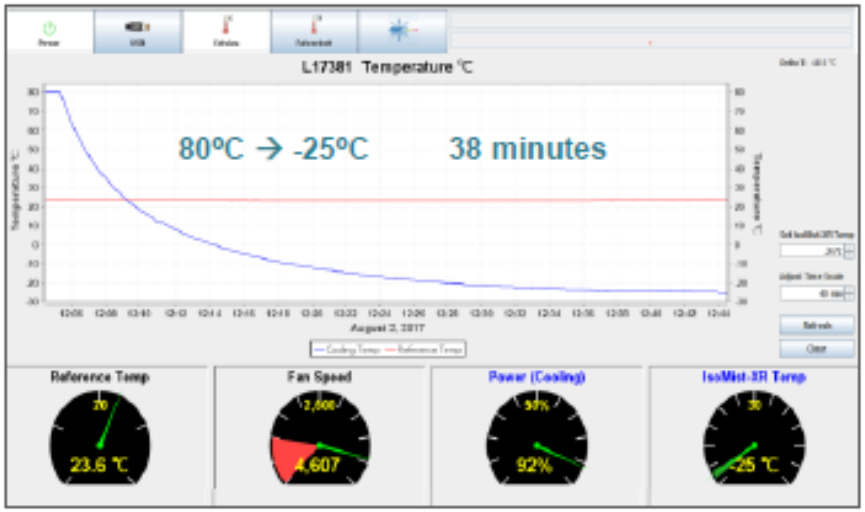
- V-groove neb
- Cooled spray chamber
- Small bore center tube

Chemical resistant pump tubing

Isomist XR





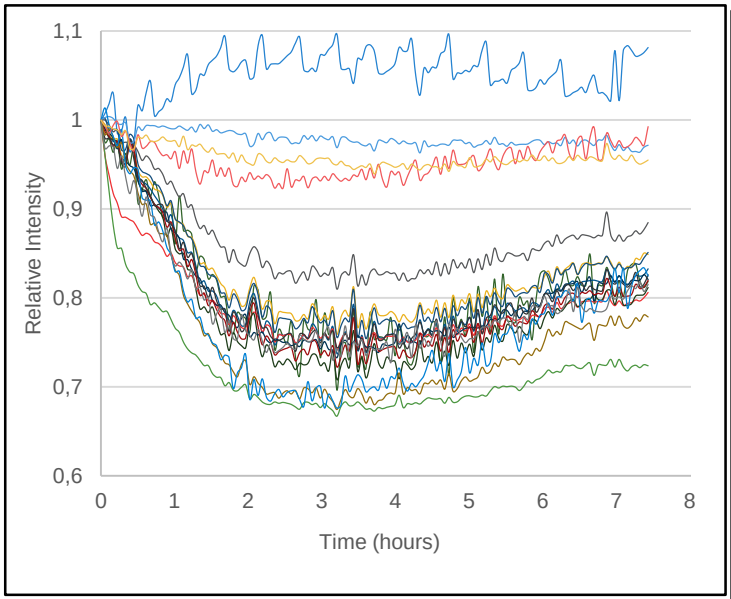


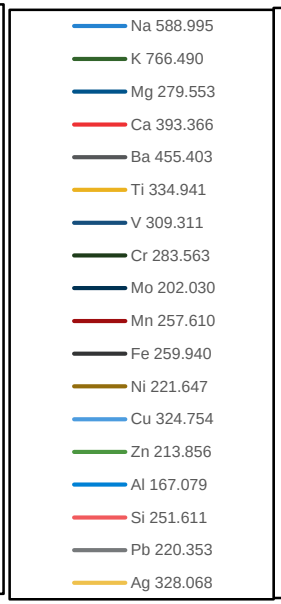
ThermoFisher SCIENTIFIC

Analysis of Naphtha with IsoMist XR

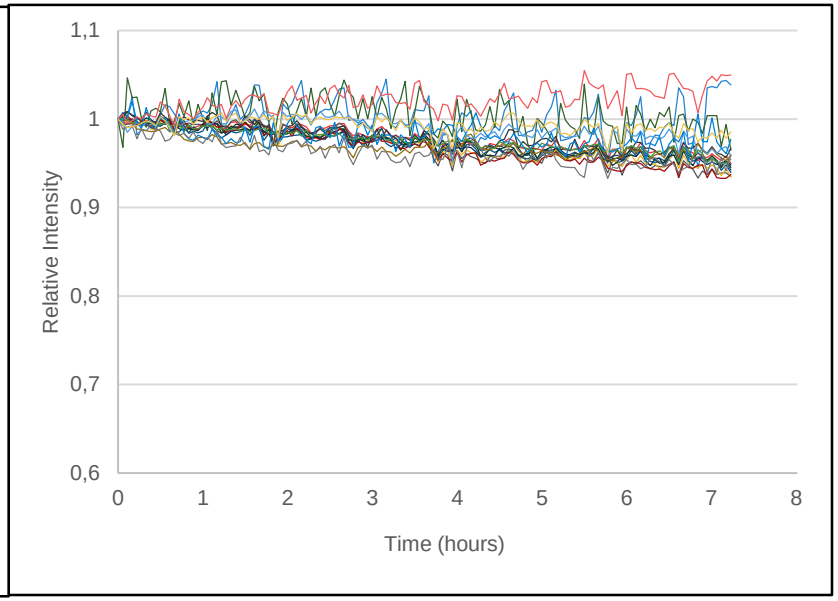
- IsoMist XR
  - Temperature range -25 °C to +80 °C

- 8h stability measurement without temperature control

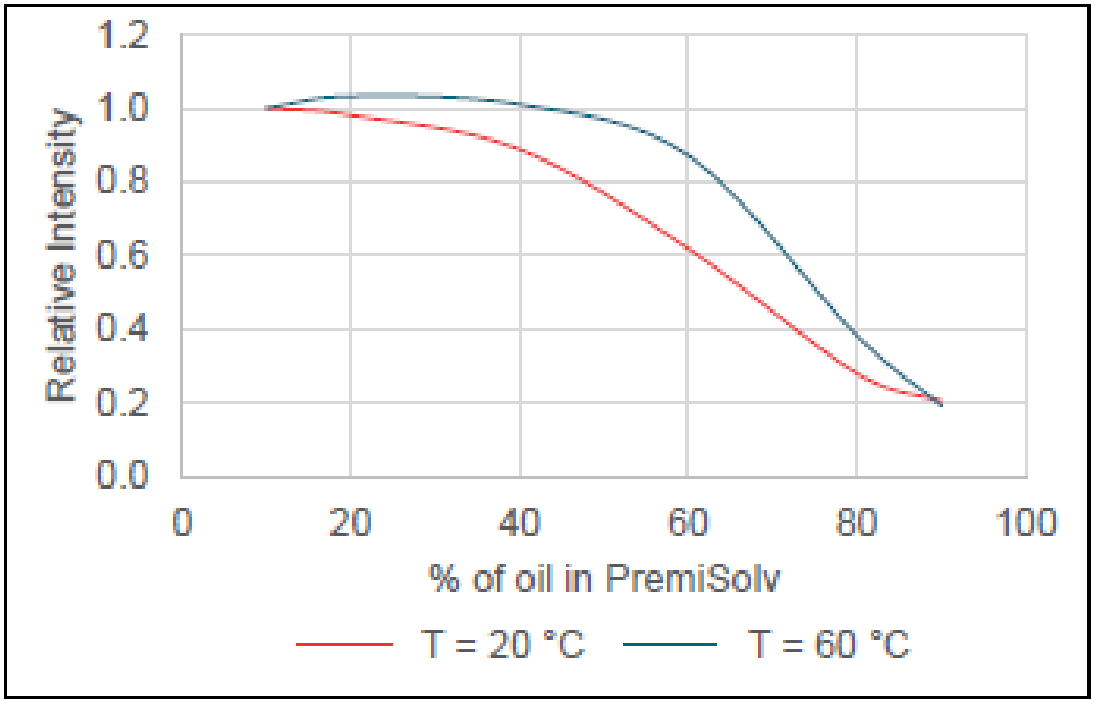




- 8h stability measurement controlled at -20°C



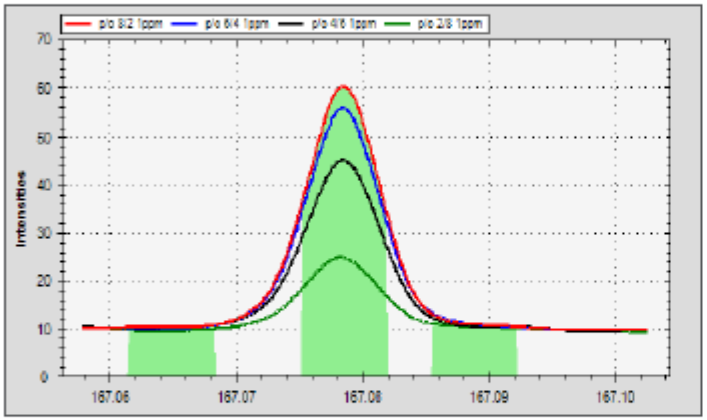
Isomist XR - heating

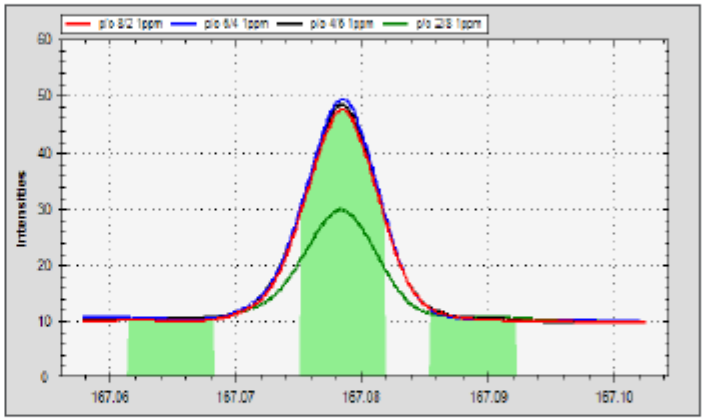


By increasing the temperature it is possible to reduce the viscosity of samples and therefore to enhance signals in ICP-OES measurements (Figure 7).

Content of oil in sample

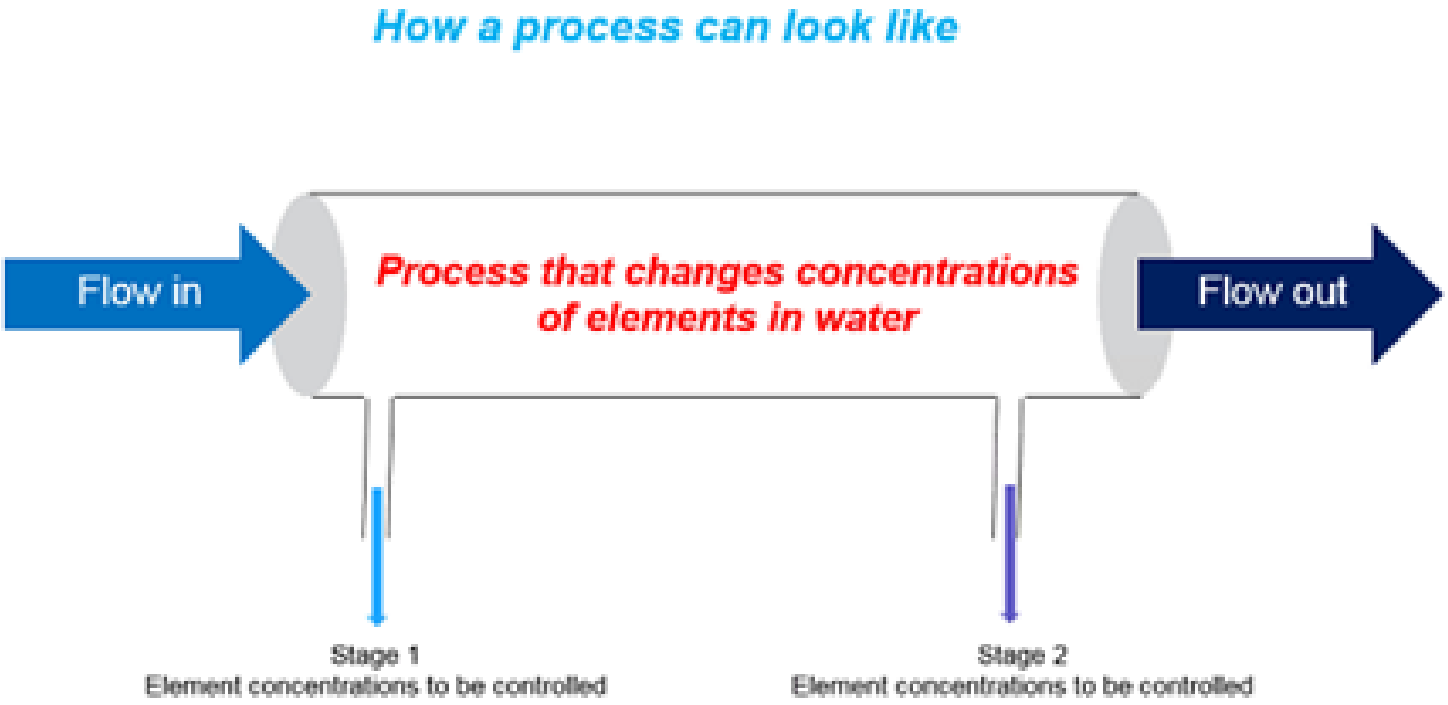
Average relative intensities for 1 mg·kg^{-1} aluminum at wavelength 167.079 nm, with pre-set temperatures at 20°C (left) and 60°C (right), depending on the content of oil in PremiSolv.





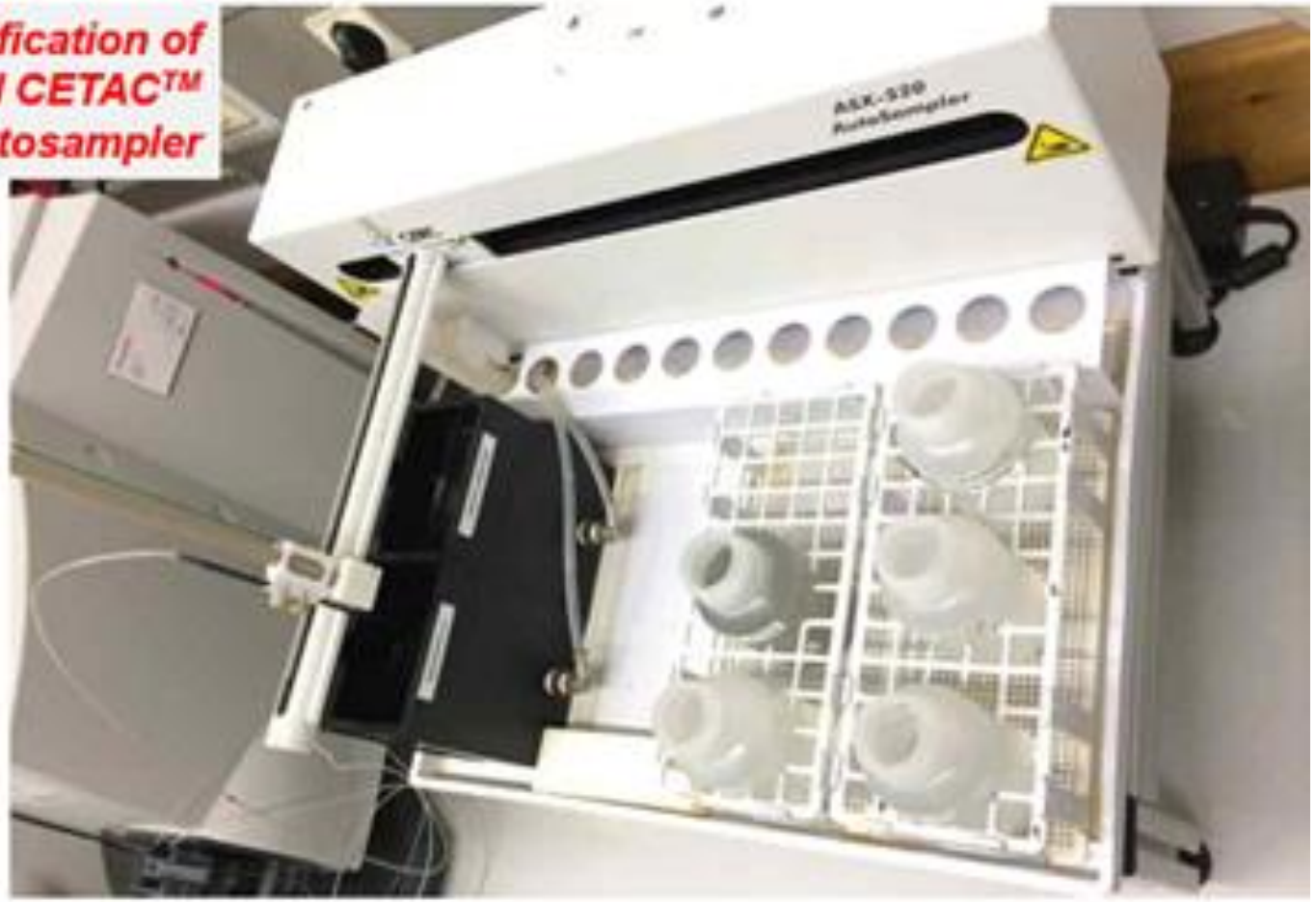
ThermoFisher SCIENTIFIC

Online sample analysis



Sewage process monitoring – 2 stages

modification of a standard CETAC™ ASX-520 Autosampler

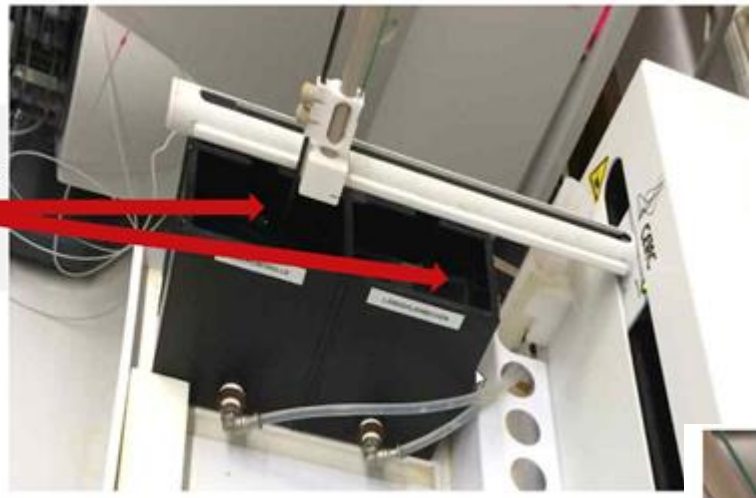


ThermoFisher SCIENTIFIC

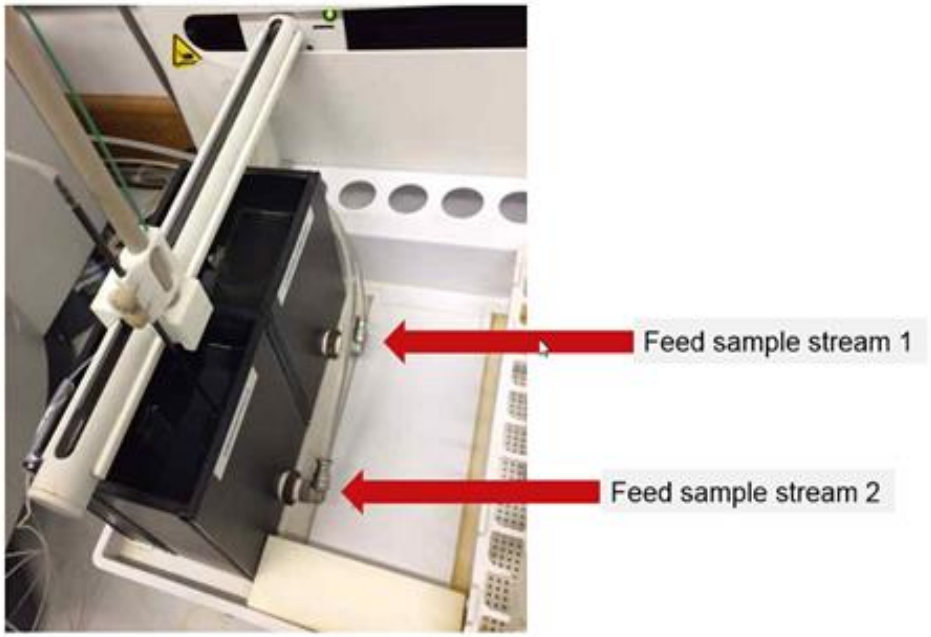
Continuous control of elements in water online

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

Feed sample analysis

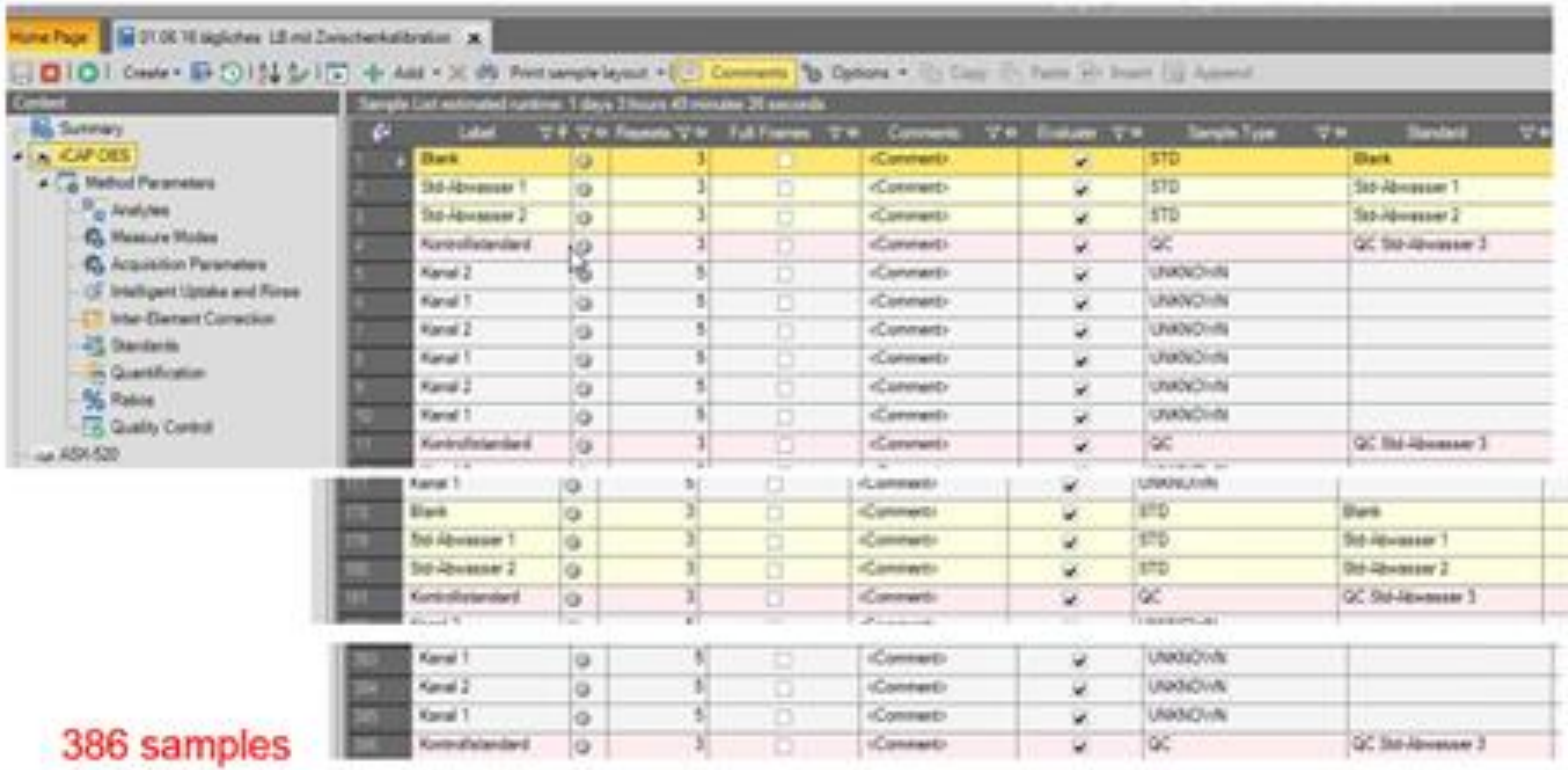


- Algae growth
- Internal standard
- Pump tube wear
- Particles in sample
- Rinse station flow



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



386 samples

in 1 days 3 hours 49 minutes

Summary

- 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.

Questions