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Method Optimization for the ICP-OES Analysis of Challenging Environmental Samples from Wastewater to Electronic Wastes

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Environmental Sample Analysis

- **Challenges when analyzing environmental samples**
  - Difficult matrices
  - Sample variety
  - Regulation and Compliance
  - Failed sample/QC analyses, subsequent re-runs
  - Sample throughput demands
  - Fast turnaround of sample/reporting results
  - Maintenance & troubleshooting
  - Training new users

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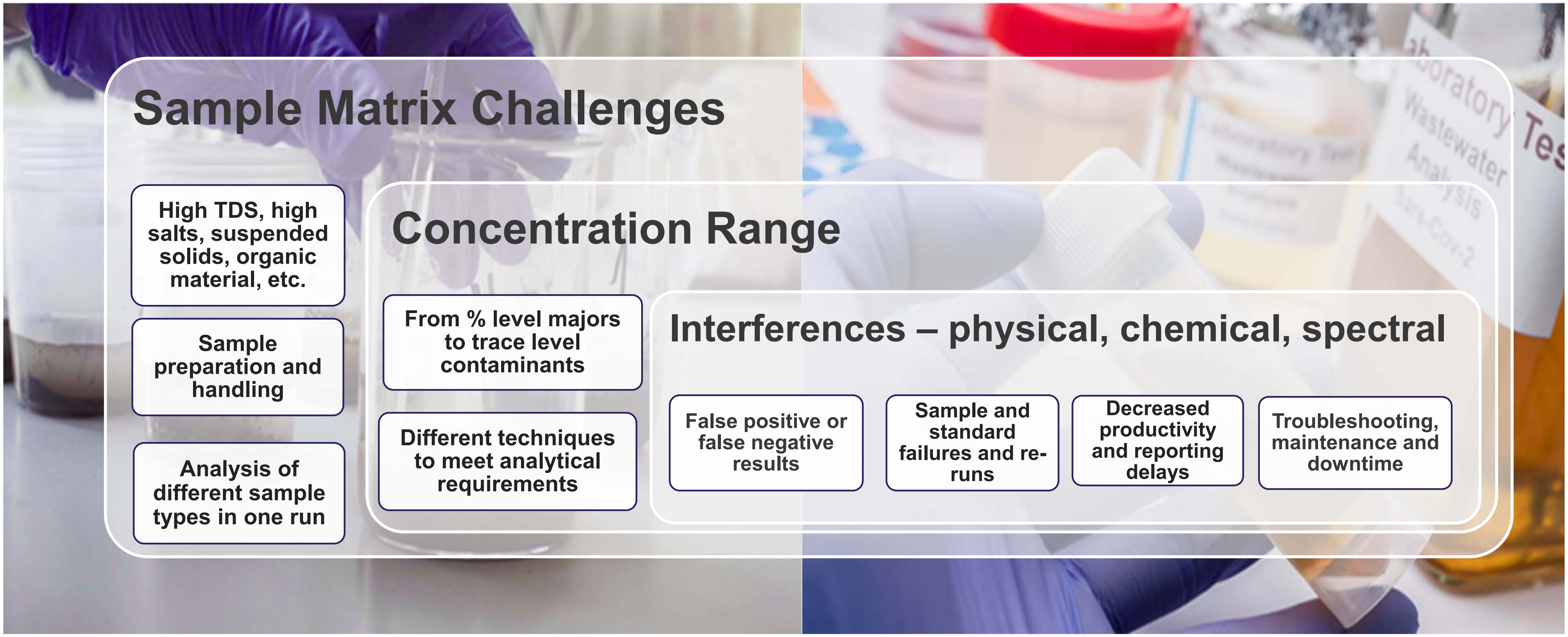
Environmental Sample Analysis

- Why are we experiencing these challenges?
- Where do we begin to address these challenges?
- How can we prevent these challenges?
- When do we call service or applications support?

➢ **Let's begin with the sample matrix…**

Environmental Sample Analysis

Layers of challenges related to sample matrix



Environmental Sample Analysis

Regulation and Compliance - added layer of challenge

- Detection limit requirements
  - National Primary Drinking Water Regulations
  - National Secondary Drinking Water Regulations
  - Unregulated Contaminant Monitoring Rule (UCMR)
  - Different state/municipal regulations
- Analysis according to EPA approved methods
  - Specific quality control protocols
    - Method validation
    - QC standards and samples
    - Control limit criteria
- Data management and transfer to LIMS
- Reporting requirements
- Data audit
- Onsite audit

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Environmental Sample Analysis

Address key challenges through

- **Instrument innovations**
  - Hardware design
  - Software features
- **Method optimization**
  - Sample introduction system
  - Plasma parameters
  - Interference correction
- **Best practices**
  - Sample handling
  - Contamination prevention
- **Troubleshooting tips**
  - Troubleshoot failures due to sensitivity, accuracy, precision and carryover issues







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Instrument Solutions For Environmental Analysis

A Portfolio of Innovative Instruments for Simplified Environmental Analysis

Atomic Absorption Spectrometry (AAS)

Flame

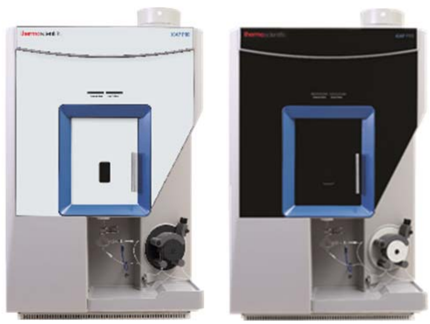
Graphite Furnace

Flame & Graphite Furnace

Thermo Scientific™ iCE™ 3000 Series AAS

- ✓ Lower investment and complexity
- ✓ GFAA offers high sensitivity for key elements
- ✓ Flame offers fast, single element analysis

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)



Thermo Scientific™ PRO™ Series ICP-OES

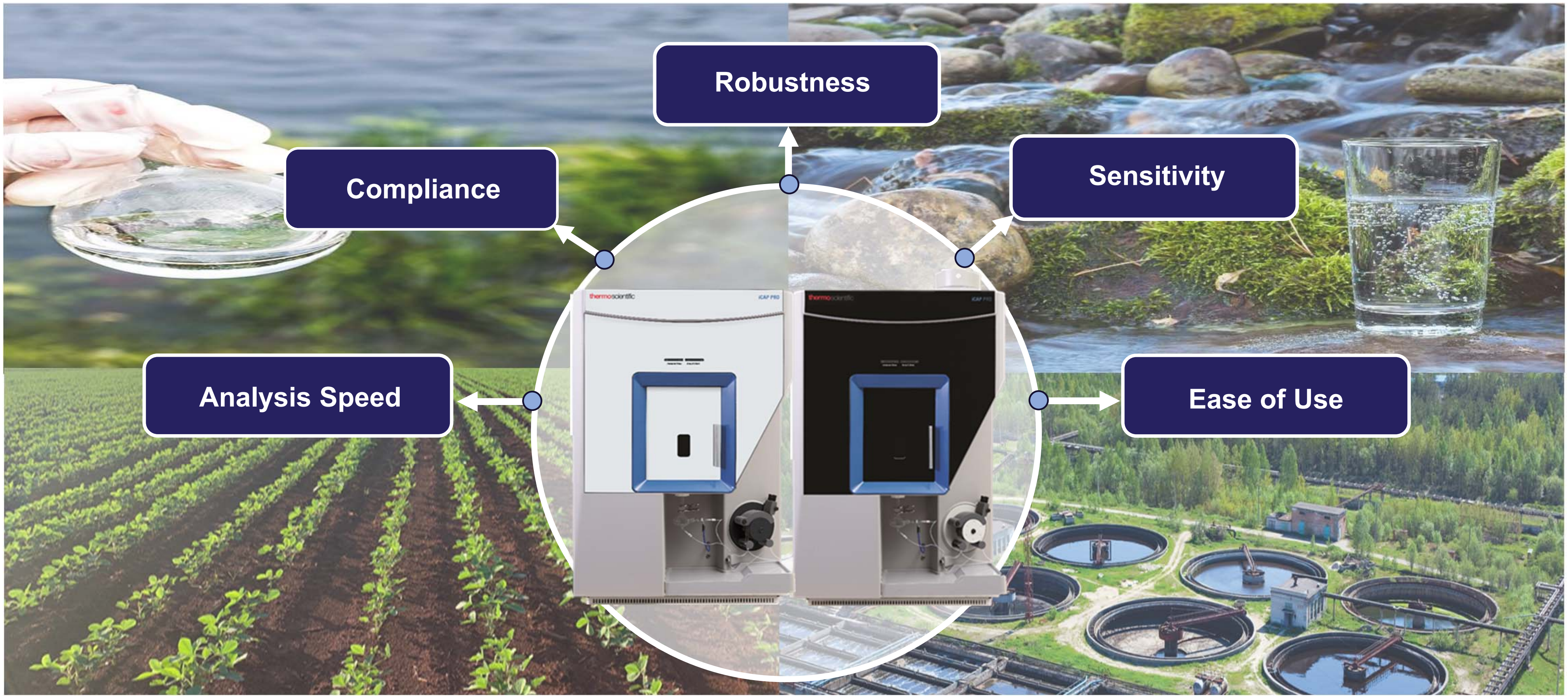
- ✓ Fast, multi-element analysis
- ✓ Robustness for high matrix samples
- ✓ Flexibility, performance and ease of use

Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

Thermo Scientific™ iCAP™ RQ ICP-MS | **Thermo Scientific™ iCAP™ TQ ICP-MS**

- ✓ Improved detection capability
- ✓ Wide linear dynamic range
- ✓ Advanced interference removal
- ✓ Speciation with chromatography

Thermo Scientific iCAP PRO Series ICP-OES



Four instrument models designed to meet the challenges in environmental analysis

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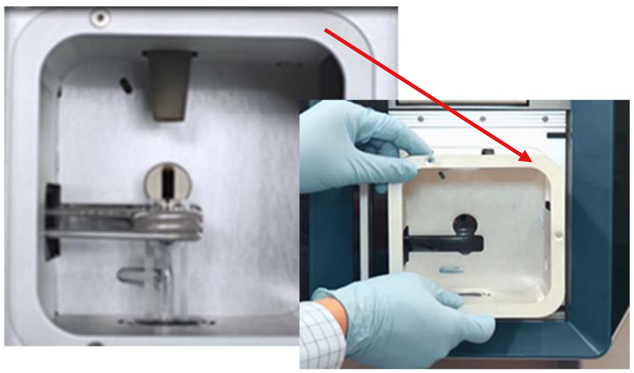
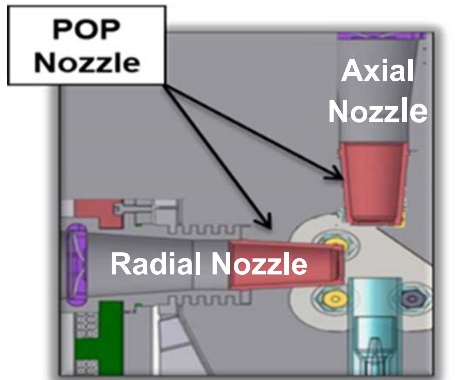
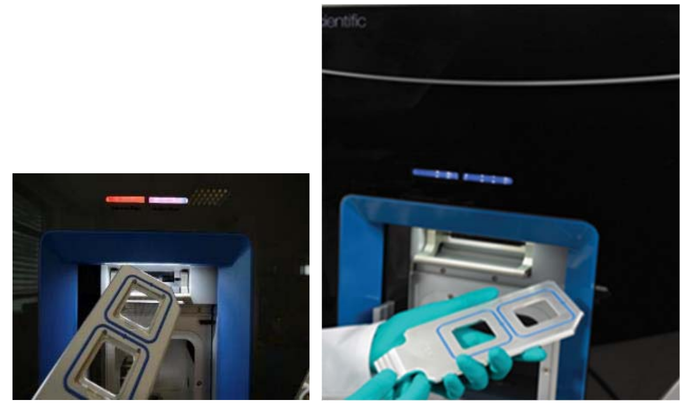
Thermo Scientific iCAP PRO Series ICP-OES

Key features for enhanced robustness for high matrix samples







Vertical Torch & Inner Torch Box

- Designed for robustness, corrosion prevention and low maintenance
- Proprietary exhaust flow for plasma stability and minimal sample deposition on torch and injector
- Inner torch box easy to remove for cleaning when necessary

Purged Optical Path Interface

- Purge gas exits optical system through axial and radial Purged Optical Path (POP) cones/nozzles removing constituents that can affect sensitivity
- Ceramic POP cones - durable, temperature and corrosion resistant

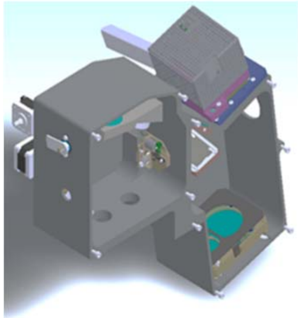
Purged Optical Path Window

- New POP window protects fore optics from dust, dirt and contamination from the plasma interface
- Easily accessed to remove for cleaning when necessary

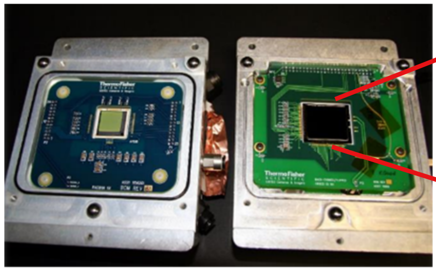
Thermo Scientific iCAP PRO Series ICP-OES

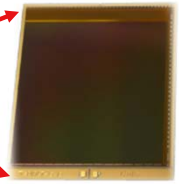
New optics and detector for enhanced speed and sensitivity

New, high-energy Echelle polychromator, side-by-side arrangement of prism and grating



CID 86
iCAP 7000
Series ICP-OES





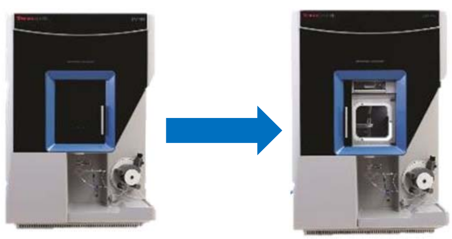
CID 821
iCAP PRO Series
ICP-OES

Optical System	CID 821 Detector
• Compact for maximum light throughput, efficiency, speed and sensitivity • Fast purge and low purge gas consumption • New Intelligent Full Range (iFR) mode for measurement of full spectrum in one exposure • New Enhanced UV (eUV) mode for higher sensitivity for key elements (e.g., As, Se, P, S) in the low UV range	• New CID 821 detector, larger chip, more pixels (over 4M pixels) compared to CID 86 • Reduced order overlap • Ultra-fast signal readout, 30-40% faster • No pre-exposure required • Continuous wavelength coverage • Immunity from blooming

Thermo Scientific iCAP PRO Series ICP-OES

Features for simplicity and ease of use

New Features for Ease of Use



Door Closed Door Open

- New, front sliding door for easy access to torch interface

- New LED instrument status panel
- Integrated torch box interface light

Small Footprint & Easy Servicing

- 24.2” (L) x 27.2” (W) x 36.7” (H)
- Small footprint of any ICP-OES
- Easy access, recessed connections
- Can be pushed close against the wall

Simplified Sample Introduction

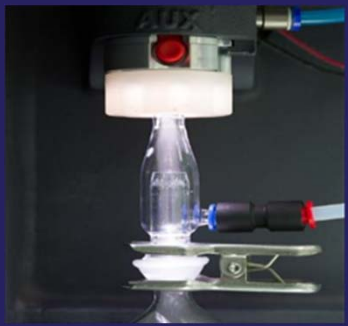
- Clip-in components, no small parts
- Quick connect torch
- Automatic gas connections
- Drain sensor for safety
- Compatibility of parts with the previous model (Thermo Scientific™ iCAP™ 7000 Series ICP-OES)

Overcoming Challenges Through Method Optimization

Four Key Areas For Method Optimization

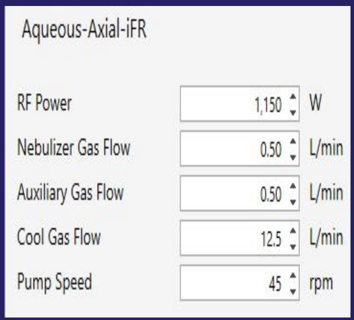
Sample Introduction System

Selection of the appropriate components is key for method optimization.



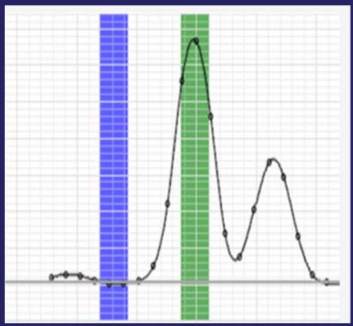
Accessories

Accessories available to improve sample handling, robustness and stability.



Operating Parameters

Set up operating parameters based on sample matrix and productivity requirements.



Interference Correction

Apply the appropriate correction techniques for physical , chemical and spectral interferences.

Sample matrix is a major consideration for optimization

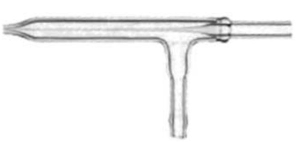
Method Optimization

Sample Introduction System

Aqueous Samples

Low solids < 3%

- Concentric glass nebulizer
- Glass Cyclonic spray chamber



High solids

> 3%

- High Solids Nebulizer (e.g., Aerosalt)
- Cyclonic chamber
- Argon humidifier

> 15%

- Parallel path nebulizers (e.g., Burgener Mira Mist)
- Baffled cyclonic spray chamber
- Sheath gas
- Large bore injector (e.g., 2.0 mm)

For enhanced robustness when running high solid samples for long periods, use a Ceramic D-torch and Sheath Gas

HF Containing

- HF resistant nebulizer (e.g.,Teflon Burgener Mira Mist)
- PFA spray chamber
- Alumina injector

Method Optimization

Sample Introduction System

Organic Samples

Non-volatile

e.g., Kerosene, Xylene, MIBK, Toluene

- V-groove nebulizer
- Baffled cyclonic spray chamber
- Small bore (1 mm ID) center tube
- Chemical resistant pump tubing

Ceramic D-torch for analysis of non-volatile organic samples, e.g., lubricating oils, for long periods

Chemical resistant peristaltic pump tubing!

Volatile

e.g., Petrol, Benzene, Hexane, Naphtha

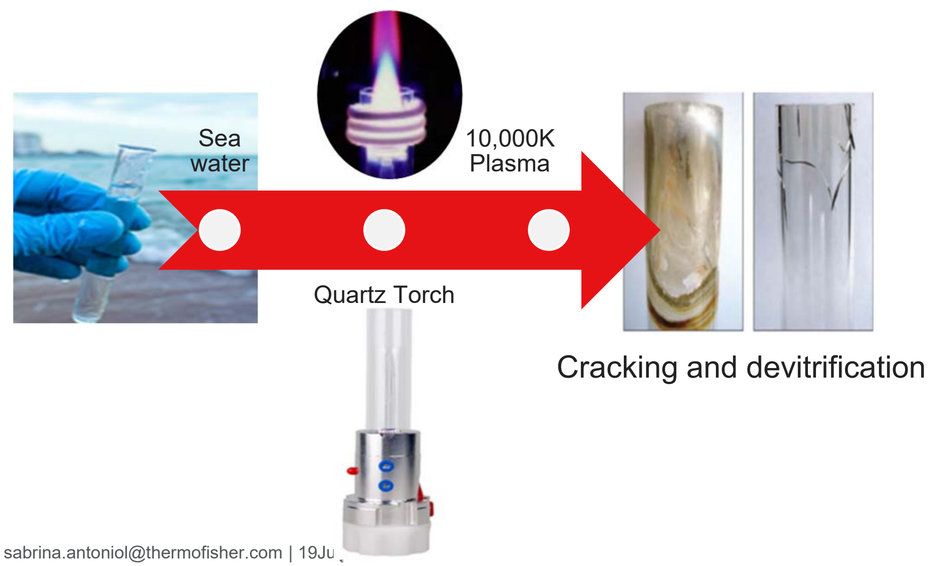
- V-groove nebulizer
- Small bore (1 mm ID) center tube
- Chemical resistant pump tubing
- Temperature controlled (cooled) spray chamber

Temperature controlled spray chamber using the **Glass Expansion IsoMist™** (operating range -10^{o}C to 60^{o}C) and the new **Glass Expansion IsoMist XR™** (extended range, from -25^{o}C to 80^{o}C)

Method Optimization

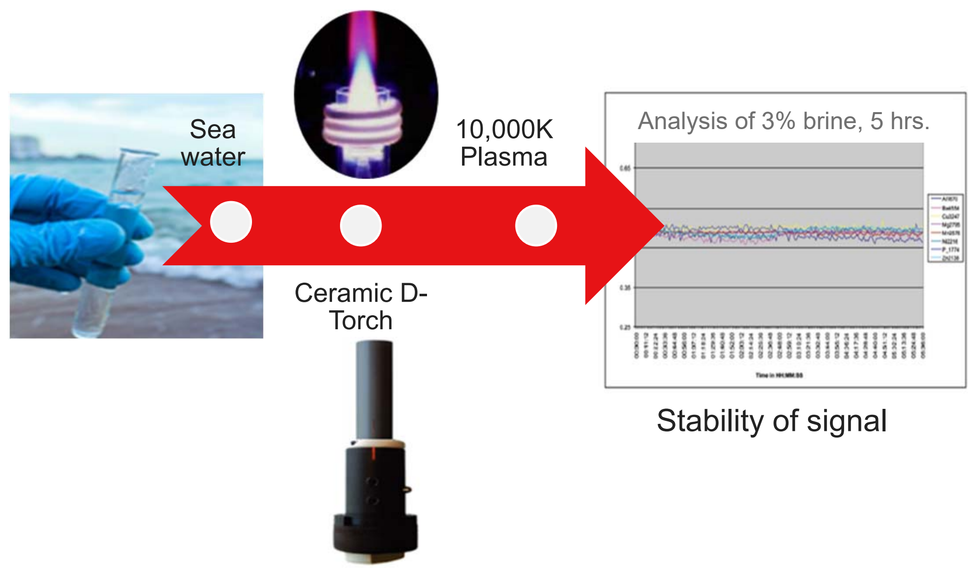
Enhanced Matrix Tolerance (EMT) Quartz Torch

- Made from Quartz, a crystalline form of SiO_2, ideal for most aqueous samples, dilute acids
- Limitation:
  - With continuous analysis of high matrix samples (e.g., sea water) quartz can devitrify/crack leading to signal instability, failed samples/QC and more maintenance



Ceramic Demountable Torch (D-torch)

- Made from Sialon (silicon nitride), a highly durable material, heat and chemical resistant material
- Alumina intermediate tube for excellent chemical and temperature resistance
- Use for high matrix samples (e.g., brines, sea water, fusions, lubricating oils, etc.,)



Method Optimization - Sample Introduction Kits

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Pre-configured kits simplifies the selection of sample introduction components

Note: Peristaltic pump tubing not included

Aqueous Kit

- Cyclonic spray chamber
- Concentric nebulizer
- 1.5 mm injector (Duo), 2.0 mm injector (radial)
- EMT torch and holder, Ball joint and clip

HF Kit

- PFA cyclonic spray chamber
- MiraMist concentric nebulizer
- 2.0 mm alumina injector
- EMT torch and holder
- Ball joint and clip

High Solids Kit

- Baffled cyclonic spray chamber
- Aerosalt concentric nebulizer
- 2.0 mm injector
- EMT torch and holder
- Ball joint and clip
- Option: Argon humidifier

Organics Kit

- Baffled cyclonic spray chamber
- V-groove concentric nebulizer
- 1.0 mm injector, EMT torch and holder
- Ball joint and clip

Volatile Organics Kit

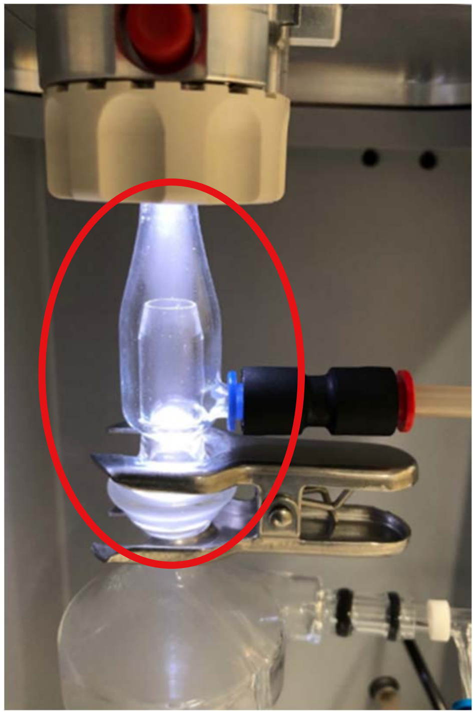
- Baffled cyclonic spray chamber
- V-groove concentric nebulizer
- 1.0 mm injector, EMT torch and holder
- Ball joint and clip
- Option: IsoMist Spray Chamber

Method Optimization - Accessories

Sheath Gas Adaptor – accessory for enhanced robustness and long-term stability

- A Sheath Gas is a constant flow of argon that envelopes the sample aerosol tangentially to
  - prevent contact with the injector
  - reduce sample deposition in the injector
- The Sheath Gas is introduced between the spray chamber and torch with the Sheath Gas Adaptor
- Benefits of a Sheath Gas
  - Enables higher tolerance of TDS
  - Less sample dilution, hence improved MDLs
  - Improvement in stability for the long-term analysis of high solid samples (e.g., sea water)
  - Reduced need for extended rinse time between samples

Sheath Gas Off



Sheath Gas On

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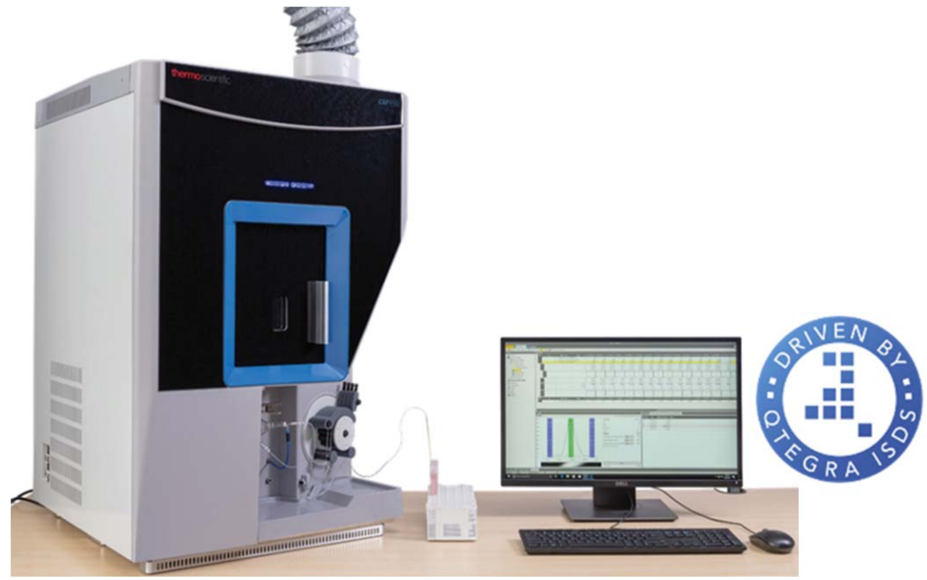
Method Optimization – Operating Parameters

Operating parameters set-up through instrument 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
  - Plug-ins for fast autosamplers and autodilution systems
  - A range of new software features (e.g., Plasma TV, auto tunes, modes, etc.) added for ease of use
  - 21 CFR Part 11 compliance tool set
  - Same software platform as Thermo Scientific ICP-MS instruments for easy cross-training



Method Optimization – Operating Parameters

Operating Parameters for Aqueous Samples

New Plasma TV
For diagnostics and optimizing instrument parameters

Operating Parameters for iCAP PRO XP and iCAP PRO XPS Duo instruments

	RF Power (Watts)	Nebulizer Flow (L/min)	Pump Speed (rpm)	Auxiliary Flow (L/min)	Coolant Flow (L/min)	Radial View Height (mm)
Range	750 – 1600 W	0 - 1.5	0 - 125	0 - 2.0	0 – 20.0	6 – 18 mm
*Aqueous Settings	1150	0.50	50	0.5	12.5	10

*standard set-up for aqueous samples, optimization needed for high TDS samples

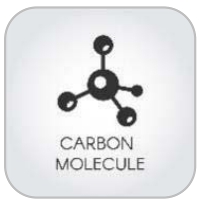
Method Optimization – Interferences

What are the interferences in ICP-OES Analysis?

Three Types of Interferences

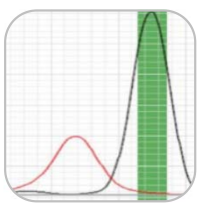
Physical Interferences

Difference in physical properties between samples and calibration standards affecting sample transport and nebulization efficiency.



Chemical Interference

Difference in the way sample and calibration standards react in the plasma during vaporization, atomization and ionization.



Spectral Interference

Characterized by an overlap of a constituent wavelength on the analyte wavelength. Also includes background signal interferences.

Method Optimization – Physical Interferences

Addressing Physical Interferences

Cause

- High TDS, suspended solids, high salts, viscosity, density, vapor pressure or volatility

Effect

- Suppression or enhancement of signal
- Instability of signal, drift during analysis
- Sample and standard failures
- More frequent maintenance

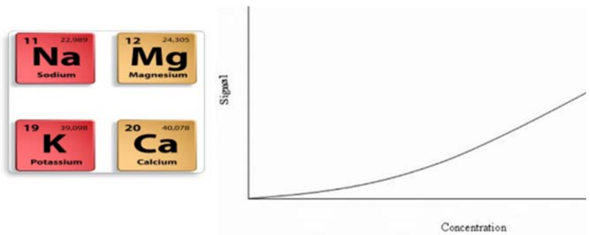
Solution

- Dilution – easiest/preferred solution
- Matrix Matching – samples and calibration standards
- Internal Standardization – online (preferred) or manual addition of internal standard
- Optimize sample introduction and operating parameters
- Method of Standard Additions – least preferred solution

Method Optimization – Chemical Interferences

Addressing Chemical Interferences

Easily Ionized Element Effect



$Na \rightleftharpoons Na^+ + e^- + e^-$ (Na, Ca, Mg, K)

- High concentration of Grp. I & II elements, excess electrons shifting equilibrium in the plasma
- **Effect:** Enhancement of atomic lines
- Solutions: radial viewing, ionization buffer (e.g., Cs, LiCl, etc.), dilution

Molecular Formation

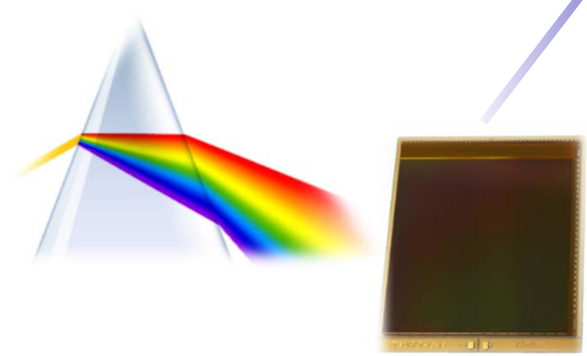
- Caused by molecular emissions in the plasma interfering with the analyte wavelength
- **Effect**
  - Elevated background
  - Spectral interferences
    - emission from carbon-containing molecules interfering with the Na 589.592 nm line
- Solutions: radial viewing, proper Background Point placement, dilution

Plasma Loading

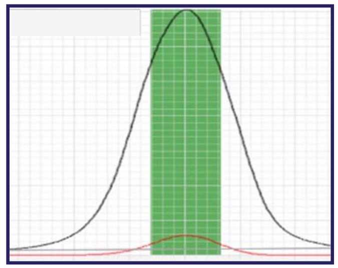
- Increased consumption of plasma energy needed to break-up high matrices (e.g., TCLP extracts) causing insufficient energy to excite low concentration or high ionization potential analytes
- **Effect**
  - Suppression of ionic wavelengths
  - Low sensitivity for key elements (e.g., As, P, S) and atomic wavelengths
- Solutions: dilution, robust plasma conditions (e.g., higher power setting, higher plasma gas flow, etc.)

Method Optimization – Spectral Interferences

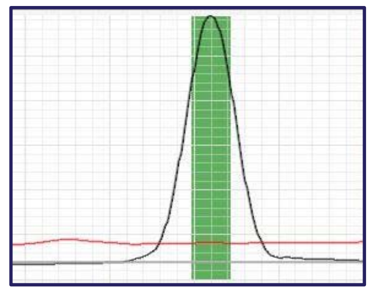
Types of Spectral Interferences



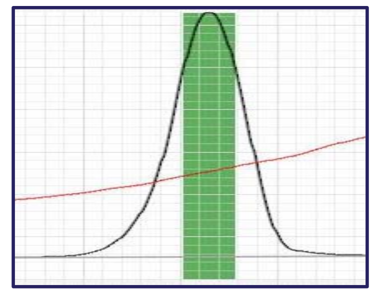
CID821 chip



Peak free from spectral interference

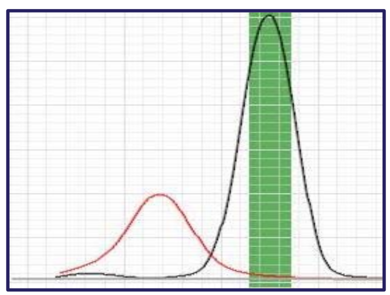


Flat baseline shift



Sloping baseline shift

Peaks with baseline shift



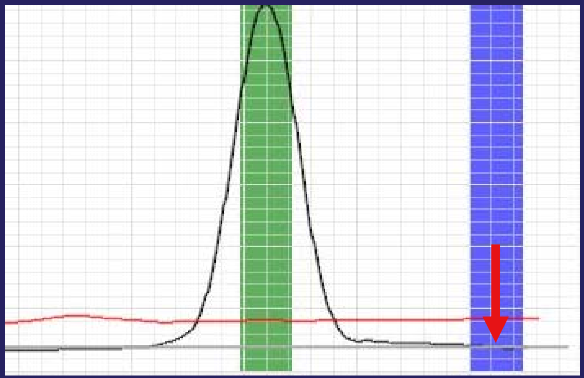
Peak with spectral overlap

Partial overlap

Method Optimization – Spectral Interferences

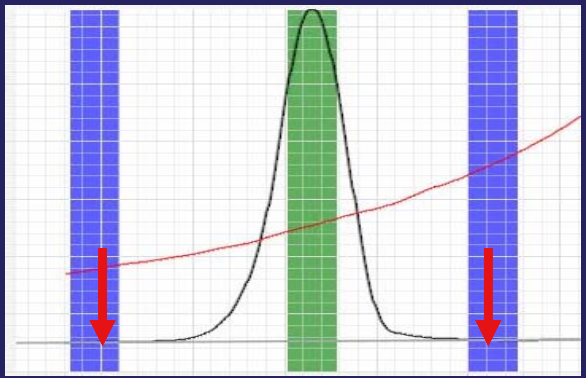
Addressing Spectral Interferences Through Background Correction

- Spectral interferences can be corrected by:
  - Applying Background Points
  - Interelement Correction Factors (IECs)



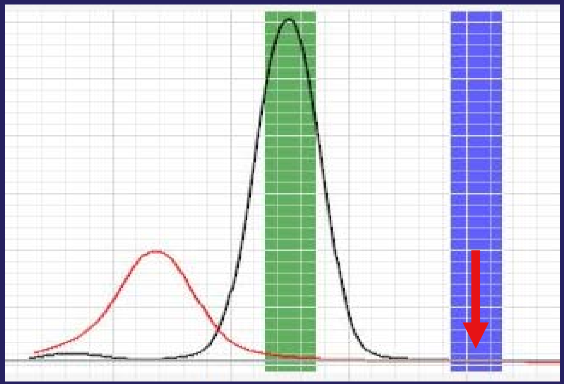
Flat Baseline Shift

- Place background point on the side of the peak with no interference



Sloping Baseline Shift

- Always use two background points on both sides of the peak



Spectral Overlap

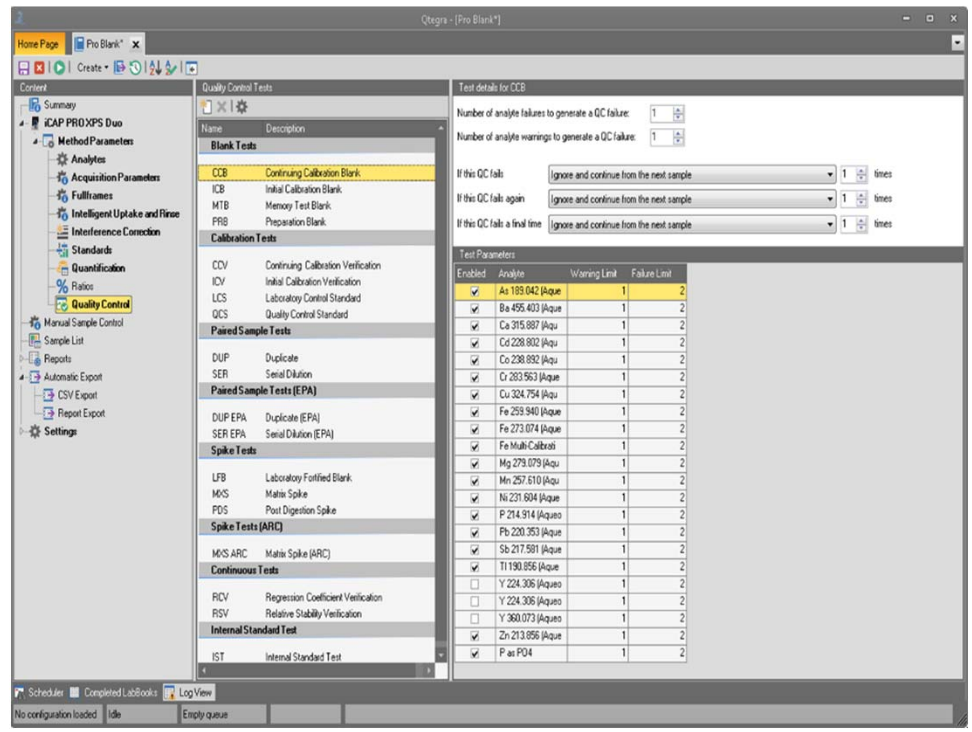
- Place background point on side of the peak with no interference
- Use alternative wavelength, if possible
- Apply Interelement Correction Factor

Method Validation

Method Validation and Quality Control – Key for Confirming Method Optimization

- **Method Validation**
  - IDL and MDL determinations
  - Good calibration curve
    - Minimum Correlation Coefficient of 0.995
  - Linear Dynamic Range (LDR) determination
  - Precision – determined by %RSD between the results of three sample replicates (short term)
  - Accuracy – confirmed by matrix spike, analysis of a Certified Reference Material (CRM)
  - Repeat above determinations over several days to confirm long-term precision and accuracy
- **Quality Control Protocol**
  - EPA QC standards and protocol built-in the Qtegra ISDS software

Qtegra ISDS Software Quality Control



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Addressing Challenges Through Best Practices

Sample handling and minimizing contamination

- Be aware of all contamination sources.
- Minimize sample handling and transfer steps.
- Use ultrapure water, high-purity acids and reagents, and certified stock standards.
- Clean apparatus using a comprehensive cleaning procedure.
- Measure weights and volumes with accuracy.
- Maintain separate sample and standard preparation areas.
- Apply proper skill, consistency, and attention to detail.

Addressing Challenges with Troubleshooting Tips

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Sensitivity, precision, accuracy, contamination, and carryover issues

Sensitivity

Sensitivity issues are typically characterized by decrease or increase of signal and failure of continuing calibration standard (CCV) recoveries.

Troubleshoot **Check the following:**

- Nebulizer or injector blockage
- Use of nebulizer appropriate for sample matrix
- Dirty spray chamber
- Sufficient purge, particularly for low UV wavelengths
- Operating parameters, nebulizer and gas flows, power setting and pump speed
- Interference and appropriate correction applied
- Old/expired calibration standards
- Analysis of second source standard for reference

Precision

Precision issues are typically characterized by high % RSD between sample replicates.

Troubleshoot **Check the following:**

- Worn peristaltic pump tubing
- Nebulizer or injector blockage
- Use of nebulizer appropriate for the sample matrix
- Dirty spray chamber
- Sufficient uptake time
- Sufficient rinse time
- Operating parameters, gas flows, pump speed
- Use of the appropriate rinse solution for sample matrix

Addressing Challenges withTroubleshooting Tips

Sensitivity, precision, accuracy, carryover and contamination

Accuracy

Accuracy issues - characterized by poor sample recoveries, failures in the analysis of QC, CRMs, and second source standards.

Troubleshoot **Check the following:**

- Nebulizer or injector blockage
- Use of nebulizer appropriate for sample matrix
- Dirty spray chamber
- Operating parameters, nebulizer and gas flows, power setting and pump speed
- Sufficient uptake time for sample matrix
- Interferences and appropriate correction applied
- Proper application of Internal Standardization
- Old/expired calibration standards

Contamination and Carryover

Contamination causes high blanks and sample/QC standard recoveries. Carryover is shown by high standard blanks (CCB) and decreasing sample replicates resulting to high % RSD.

Troubleshoot **Check the following:**

- Sufficient rinse time for sample matrix
- Appropriate rinse solution for sample matrix
- Dirty spray chamber
- Contaminated DI water supply and acids, use trace metal or higher-grade acid if possible
- For “sticky” elements (e.g., Hg, Mo, Sb), use longer rinse times. For Hg, use Au to help rinse out Hg.
- Clean work bench/environment free of dust and dirt

Optimized Methods for Environmental Samples

Application Survey

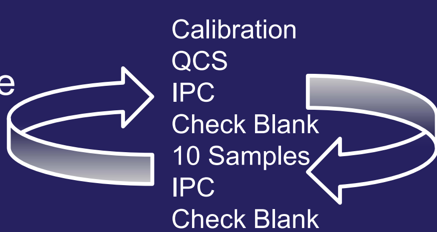
www.thermofisher.com/icappro/

Analysis of Water Samples by US EPA Method 200.7

Application Note 44422 – US EPA Method 200.7 Using the Thermo Scientific iCAP PRO XPS Duo

www.thermofisher.com/icappro/

- **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% HNO_3
  - Internal Standard: 5 mg/L Yttrium added online
  - Samples - Drinking water, trench water and well water
- **Analysis**
  - MDL and LDR Study
  - SIC solutions
  - Run sequence



152 total samples (with 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
RF Power	1150 W
Injector	2 mm
Replicates	3
Radial view height	10 mm
Exposure Time	Axial View – 10 seconds Radial View – 10 seconds

optimized for aqueous samples, <3% TDS

Analyzed using iFR mode for all 31 elements

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Analysis of Water Samples by US EPA Method 200.7

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Sample Results

- All spike sample recoveries within the required range of 85%-115%

Application Note 44422

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Analyte	Drinking Water			Trench Water			Well Water		
	Unspiked (mg/L)	Spiked (mg/L)	Recovery (%)	Unspiked (mg/L)	Spiked (mg/L)	Recovery (%)	Unspiked (mg/L)	Spiked (mg/L)	Recovery (%)
As	<MQL	0.200	100.0	<MQL	0.203	101.5	<MQL	0.203	101.5
Ca	40.52	42.43	95.5	49.92	57.39	99.6	46.20	53.66	99.5
Cd	<MQL	0.199	99.5	<MQL	0.196	98.0	0.01	0.199	99.0
Cu	0.024	0.319	98.3	<MQL	0.291	97.0	0.007	0.296	96.3
Fe	0.045	0.239	97.0	1.360	8.701	97.9	27.40	34.82	98.9
Hg	<MQL	0.196	98.0	<MQL	0.196	98.1	<MQL	0.197	98.5
K	2.747	7.795	101.0	12.56	15.31	110.0	1.401	4.116	108.6
Mg	4.271	11.60	97.7	7.863	14.95	9.45	6.953	14.02	94.2
Na	14.24	19.67	108.6	145.31	170.8	102.0	92.85	118.3	101.8
P	0.015	1.644	108.6	0.102	1.730	108.5	1.185	2.742	103.8
Pb	<MQL	0.197	98.5	<MQL	0.192	96.0	0.077	0.266	94.5
Sb	<MQL	0.200	100.0	<MQL	0.195	97.5	<MQL	0.197	98.5
Se	<MQL	0.193	96.5	<MQL	0.193	96.5	<MQL	0.197	98.5
Tl	<MQL	0.198	99.0	<MQL	0.281	93.7	<MQL	0.283	94.3
Zn	0.0009	0.22	109.6	0.0013	0.22	109.4	0.282	0.48	99.0

Analysis of 25% NaCl Samples

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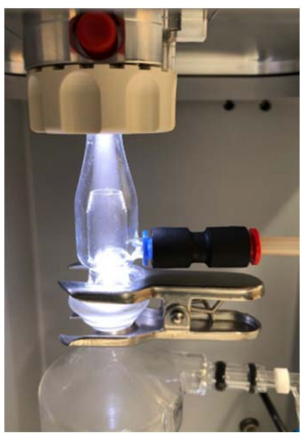
Instrumentation, Sample Introduction and Operating Parameters

- **Instrumentation**
  - Dedicated radial and duo instruments were used to compare performance and results for this high matrix sample
  - For extra robustness, the Ceramic D-torch and Sheath Gas were used



Parameter	Setting for iCAP PRO XP ICP-OES Radial	Setting for iCAP PRO XP ICP-OES Duo
Pump Tubing	Sample Tygon® orange/ white Drain Tygon® white/white	Sample Tygon® orange/ 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 $L\cdot min^{-1}$	0.55 $L\cdot min^{-1}$
Auxiliary Gas Flow	0.5 $L\cdot min^{-1}$	1.5 $L\cdot min^{-1}$
Coolant Gas Flow	12.0 L $L\cdot min^{-1}$	12.0 $L\cdot min^{-1}$
Additional Gas	0.15 L $L\cdot min^{-1}$	0.15 $L\cdot min^{-1}$
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

Optimized for high salt samples

Long wash not needed with the Sheath Gas

Sheath Gas Flow

Higher power for high salt samples

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Analysis of 25% NaCl Samples

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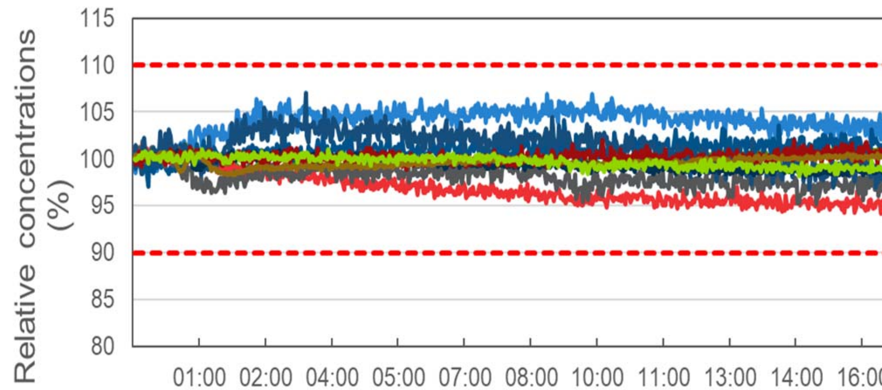
Results – Sensitivity and Long-Term Stability Tests

Method Detection Limits (MDL) obtained from different configurations of the iCAP PRO XP Duo 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})
Al 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) stability test using the iCAP PRO XP ICP-OES, Dedicated Radial instrument



Analytes – Al, Ba, Co, Cr, Cu, Fe, Mn, Ni, Sr, Zn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High matrix tolerance of the Thermo Scientific iCAP PRO XP ICP-OES Radial and Duo equipped with a ceramic torch and sheath gas adapter

Application Note 44470

US EPA 6010D (SW-846) using the iCAP PRO XP ICP-OES

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Soil and water samples

- iCAP PRO XP ICP-OES Duo used for the analysis of soil and water samples according to US EPA Method 6010D (SW-846)
- Soil and water samples digested by hot plate and microwave according to US EPA SW-846 sample preparation procedures
- Two soil Standard Reference Materials (SRM) samples analyzed for added method validation
- Teledyne CETAC™ ASX-560 autosampler combined with a Teledyne CETAC™ ASXpress™ system
- Internal standard added online, 5 mg/L Yttrium

Teledyne CETAC is a trademark of Teledyne CETAC Technologies. *ASXpress* is a registered trademark of Teledyne Cetac Technologies.

Analysis of Soil and Water Samples by Method 6010D (SW-846)

- **iCAP PRO XP ICP-OES Duo**
  - iFR mode used for analysis
  - Simultaneous analysis of wavelengths between 167-852 nm in one exposure for axial or radial views
  - Fast start-up for routine labs – 5 minutes after warm-up
  - Fast sample analysis using rapid sample introduction system accessory – 1 minute and 28 seconds analysis per sample
  - Interference correction
    - Physical interferences – online Internal Standardization
    - Spectral interferences – Interelement Correction Factors (IECs) calculated by concentration automatically within Qtegra ISDS

Parameter	Setting
Pump tubing	Sample: Tygon™ orange/white Drain: Tygon™ white/white Internal standard: Tygon™ orange/blue
Pump speed	45 rpm
Spray chamber	Glass cyclonic
Nebulizer	Glass concentric
Nebulizer gas flow	0.55 L·min^{-1}
Coolant gas flow	12.5 L·min^{-1}
Auxiliary gas flow	0.5 L·min^{-1}
Center tube	2 mm
RF power	1,250 W
Replicates	3
Sample loop	2 mL
Exposure time	Axial: 7 s Radial: 7 s

Optimized for sample matrices and for fast analysis (Pump speed through Exposure time)

Tygon is a trademark of Saint-Gobain Performance Plastics.

Method Validation - SRM Results

- Method performance can also be verified by analyzing SRMs
- NIST SRMs were digested using microwave assisted acid digestion according to EPA Method 3051A
  - All results should be within ±10% of the certified values (or as stated on the certificate)


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Fast, accurate, and robust analysis of environmental samples according to US EPA Method 6010D

Application Note 74146

Element	SRM 2781 – Domestic Sludge, NIST			SRM 2709a – San Joaquin Soil, NIST		
	Measured (mg/kg)	Certified value (mg/kg)	Recovery (%)	Measured (mg/kg)	Certified Value (mg/kg)	Recovery (%)
Al	16253	16000	102	70636	73700	96
As	8.1	7.81	104	11.2	10.5	107
Ba				992	979	101
Be	0.5820	0.6133	95			
Cd	118.3	12.78	93	0.348	0.371	94
Co				12.2	12.8	95
Cr	208	202	103	129	130	99
Cu	607.3	627.8	97	32.0	33.9	94
Fe	28357	28000	101	32508	33600	97
K	4962	4900	101	20649	21100	98
Mg	5953	5900	101	14925	14600	102
Ni	78.2	80.2	98	83	85	98
P	24722	24300	102	704	688	102
Pb	206	200.8	103	16.4	17.3	95
Se	17	16	106			
Tl				0.559	0.58	96
Zn				98	103	95

Analysis of REE in electronic waste

APPLICATION NOTE 44466 - Robust analysis of REE in electronic waste

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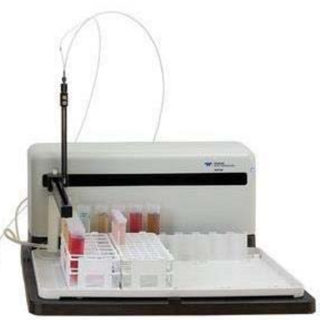
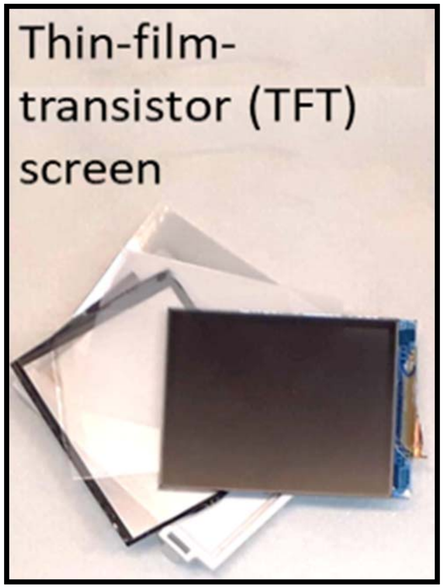
- **Why is an application on e-waste important?**
  - Massive amounts of electronic waste generated worldwide
  - High demand in modern recycling and recovery industry
  - Waste management / Environmental impact
- **Challenges**
  - Complicated and varied matrices
  - High interferences because of overlapping REEs wavelengths
  - Low concentrations of precious and poisonous elements
- **Solution**
  - Thermo Scientific iCAP PRO XP Duo ICP-OES
  - Teledyne CETAC ASX-560 Autosampler

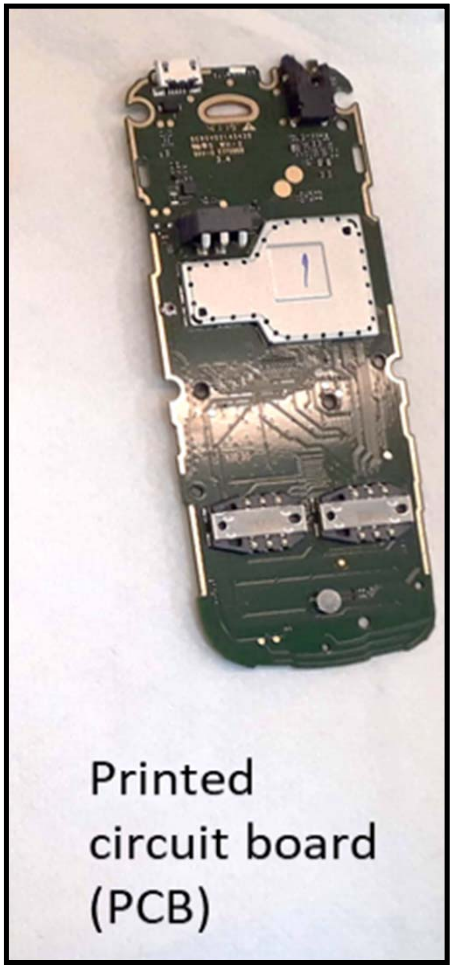


E-waste Sample Selection

Electronic waste – mobile phones

- **Components Analyzed**
  - Phone screen (TFT)
  - Circuit board (PCB)
  - Magnet from speaker
- **Analytes of Interest**
  - Rare earth elements
    - – Lanthanide series + Th, Y, Sc
  - Other metals
    - – Ag, Au, etc. (precious)
    - – Cd, Pb, etc. (toxic)





Sample Preparation and Instrument Parameters

- **Sample Preparation**
  - PCBs and TFTs ground into powders and digested using 1:1 HNO_3 : HCL
  - Magnets digested whole using Aqua Regia
- **Final Sample Solutions**
  - Filtered and brought to volume with DI H_2O to contain 10% Trace Metals Grade Acid (HNO_3 & HCl)

Application Note 44466
www.thermofisher.com/icappro/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APPLICATION NOTE 44466

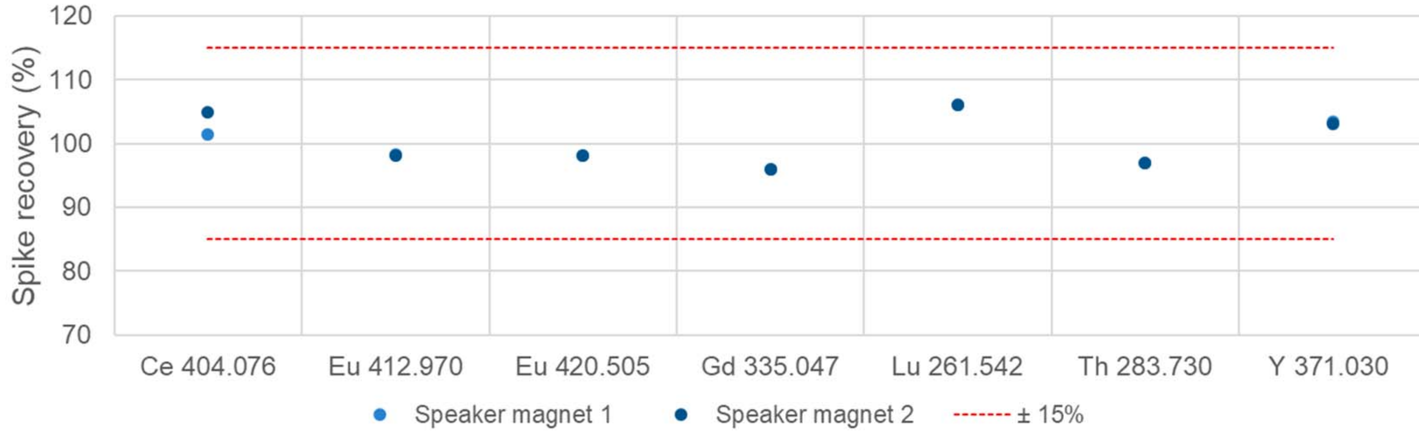
Robust analysis of REE in electronic waste

High sample throughput with analysis times of less than 2 minutes per sample with the iCAP PRO Series ICP-OES

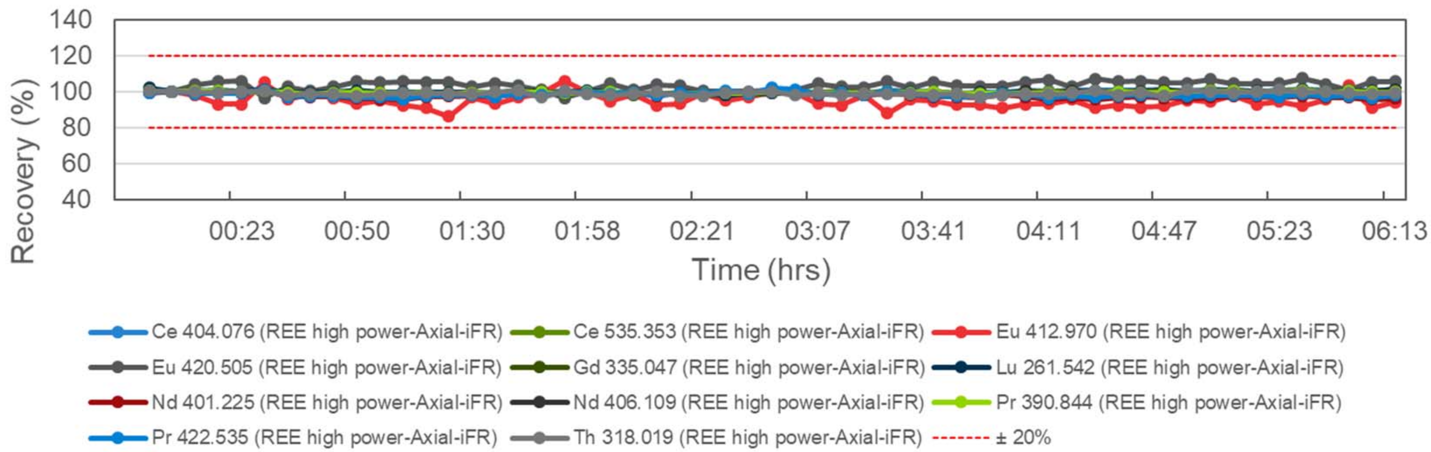
iCAP PRO XP Duo ICP-OES Parameters	
Pump Tubing	Sample Tygon® orange/ white
	Drain Tygon® white/ white
Spray Chamber	Glass cyclonic
Nebulizer	Standard glass nebulizer
Center Tube	1.5 mm (quartz)
Torch	Quartz Duo torch
Pump Speed	45 rpm
Flush Pump Speed	100 rpm
Pump Stabilization Time	10 s
Wash Time	20 s
Nebulizer Gas Flow	0.65 $L \cdot min^{-1}$
Auxiliary Gas Flow	0.5 $L \cdot min^{-1}$
Coolant Gas Flow	14.0 L $L \cdot min^{-1}$
Additional Gas	0.15 L $L \cdot min^{-1}$
RF Power	1300 W

Results

- **Accuracy**
  - Matrix Spike Recoveries of Magnet Samples



- **Robustness**
  - Long-term stability demonstrated by 6-hour analysis of simulated sample



Summary and Conclusion

Overcoming the many challenges in the analysis of environmental samples by ICP-OES starts with handling the sample matrices and variety of samples analyzed through innovative instrumentation and methods optimized for the sample matrices and the interferences they present during analysis.

Sample matrix is a key consideration for method optimization. Sample introduction system, accessories and operating parameters must be selected to handle the sample matrix. Addressing physical, chemical and spectral interferences is essential for accurate data. Finally, the Qtegra ISDS software includes new features for instrument tuning and the quality control protocol to streamline method development.

The iCAP PRO Series ICP-OES provides the robustness, sensitivity, speed, compliance tools and simplicity needed by environmental laboratories to run their samples daily. Four new iCAP PRO ICP-OES models are optimized for the sample matrix and productivity needs. **www.thermofisher.com/icappro/**