Analysis of difficult samples with ICP-OES

Agilent 5110 ICP-OES

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Agilent
Trusted Answers

Benefits of the 5100

Lowest cost of ownership

- Fastest sample throughput
- Low gas consumption

Enhanced Performance

- Analytical performance
- System robustness and reliability

Simple Operation

- Hardware
- Software

Difficult Samples

Difficult Samples

Running highly concentrated, complex samples brings a range of new challenges:

Solid Build Up in Torch Injector

- Changes gas flow velocity and observation zone. This manifests as drift.

Solid build up in Nebulizer

- Blockage leads to poor precision and signal drift.

Organic solvent based samples

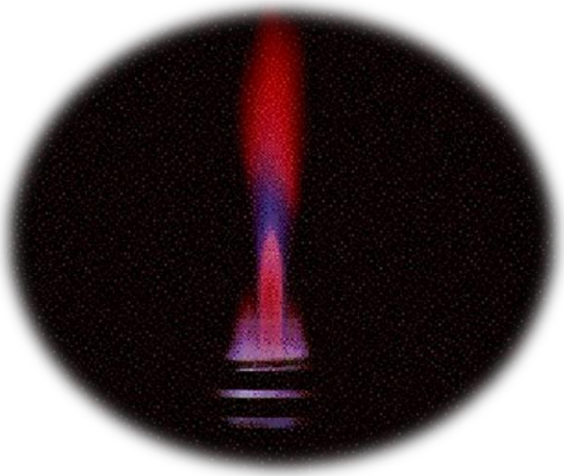
- Precision can be impacted.

Interferences from the sample matrix on the analytes

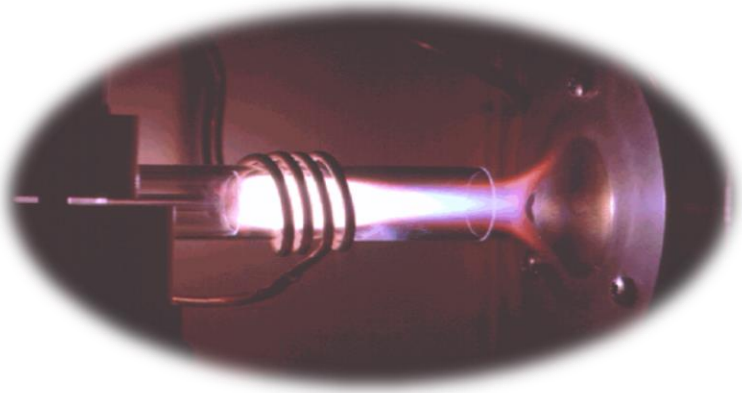
- Results accuracy and truness can be impacted

Difficult Samples

Samples with high levels of dissolved solids can start to deposit in the injector.



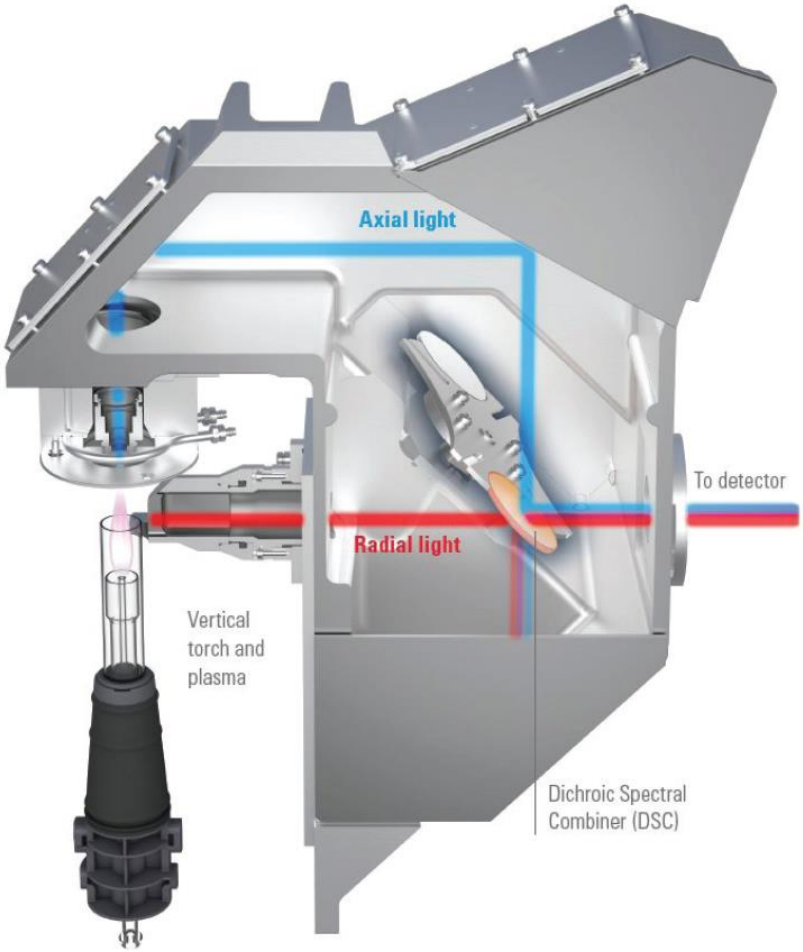
Vertical Torch – 25% TDS capability



Horizontal Torch – 5% TDS capability

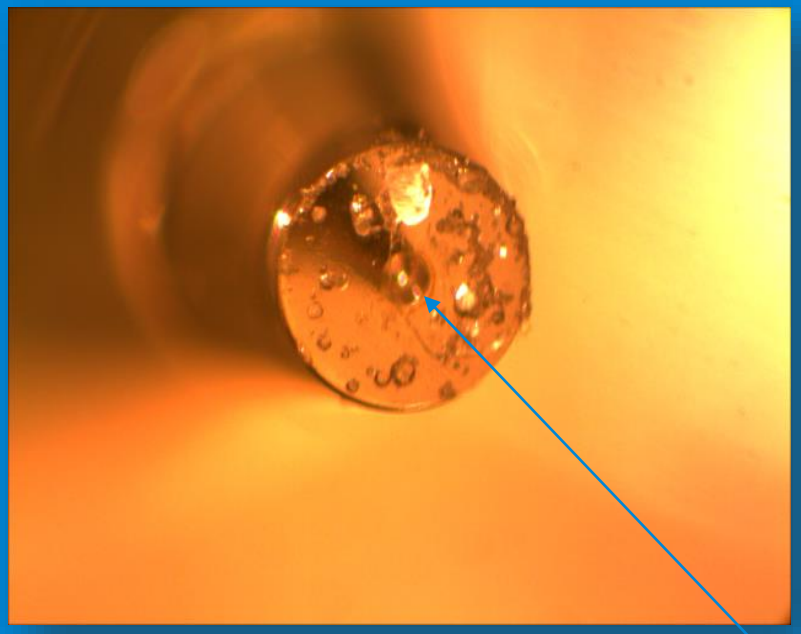
Difficult Samples

5110 ICP-OES series : Vertical Torch System and Dual View capacity



- Vertical torch for matrix tolerance.
- Axial view for high sensitivity.
- Radial view for robust measurement (EIEs).
- DSC technology allows axial and radial light to be detected simultaneously.

Difficult Samples



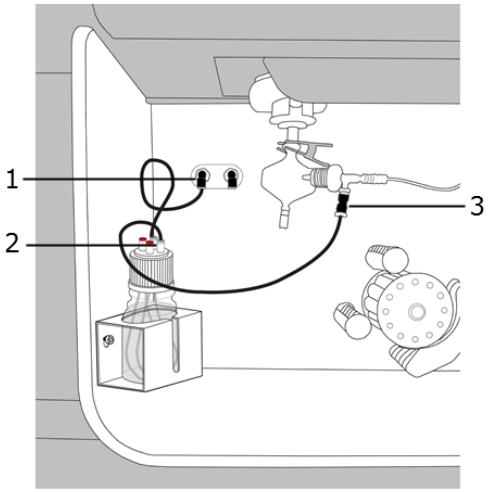
- Desolvation of high TDS sample occurs due to the fast flowing gas through the small nebulizer orifice – leads to crystalline material depositing
- High Total Dissolved Solids samples can lead to poor precision.

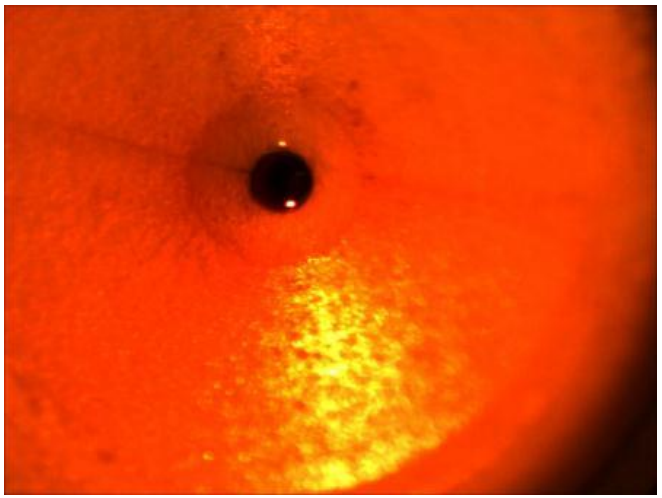
White salt crystal lodged in nebulizer orifice

Difficult Samples
Argon Humidifier Accessory (AHA)

- Permeable PTFE membrane allows H_2O vapor into the nebulizer gas.

- Humidifies nebulizer gas to help reduce:
  - Nebulizer blockages owing to salt build-up.
  - Long term **drift** from deposits in torch injector.



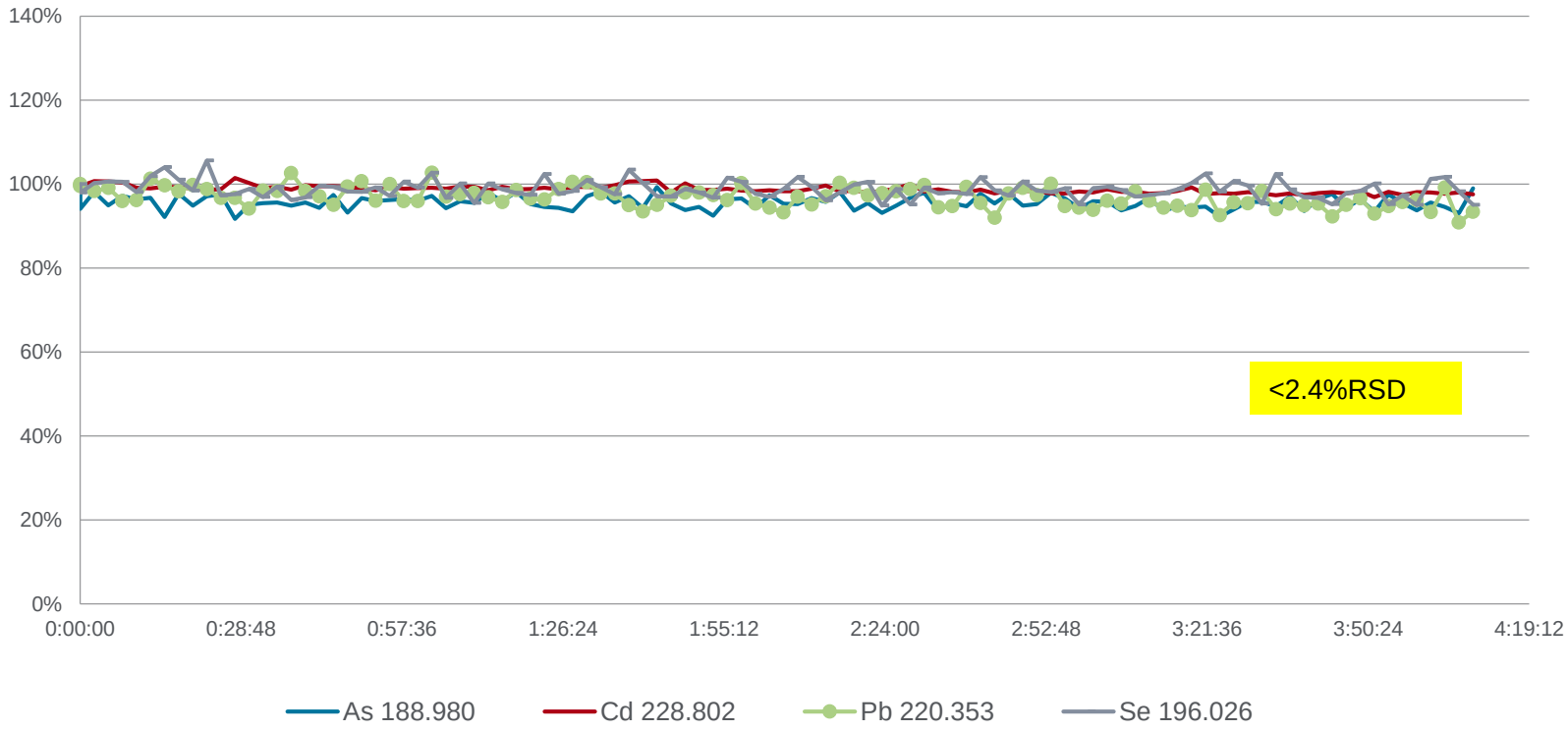


Difficult Samples
Double-Pass Spraychamber

- Double-pass spraychambers reduce sample loading on plasma:
  - Effectively sorts out larger droplets that destabilize plasma and degrade precision.
  - Reduces build-up of deposits in torch injector that can change nebulizer gas velocity through plasma central channel causing long term drift.

250 ppb Multi-element in 25% NaCl

<2.4% RSD Long Term Stability with VDV Configuration (axial only mode)



OneNeb, Double pass glass cyclonic, Blk/Blk uptake pump tubing, Ar humidifier,1.8mm injector demountable torch, 15rpm peristaltic pump

Organic Samples

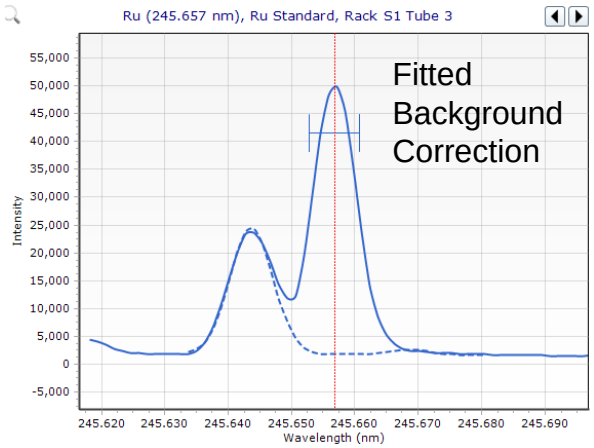
- Select appropriate sample introduction system.
- Incorrect selection leads to plasma instability – poor precision and even plasma extinguishing.
  - Typically requires a narrower Internal Diameter injector than standard e.g.
    - 1.4 mm ID torch injector for non-volatile organics such as kerosene.
    - 0.8 mm ID torch injector for volatile organics such as gasoline or naphtha.
  - Use appropriate solvent resistant pump tubing.

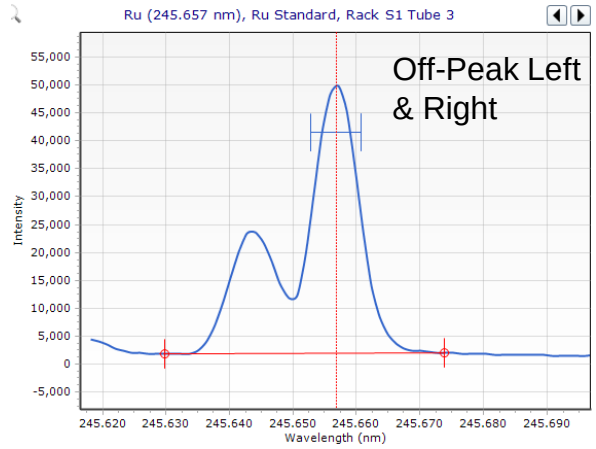
Organic Samples

- For volatile organics use a cooled spraychamber (see below) at sub-ambient conditions e.g. -10^{o}C
- Organic resistant pump tubing such as marprene, solvent flex and viton.

May need to use some oxygen addition into auxillary Ar flow to prevent carbon deposition on injector

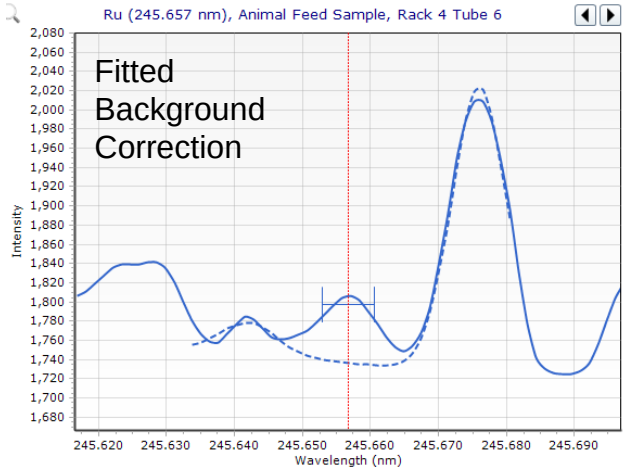
Fitted Background Correction (FBC)

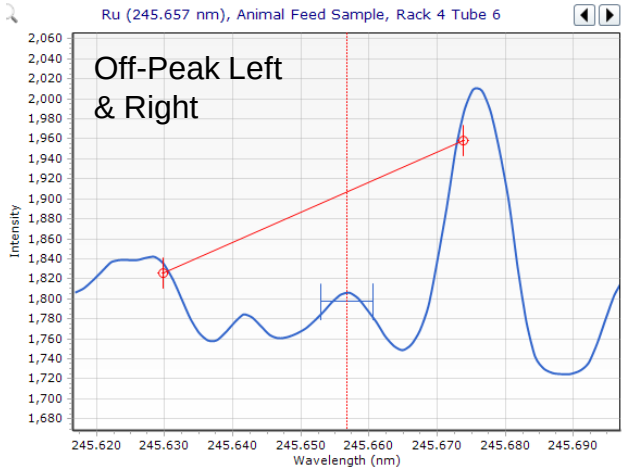




- Fully automated background correction.
- Automatically models the background signal under the analyte peak using a sophisticated mathematical algorithm.
- Traditional Off-Peak background correction requires the background points to be manually selected.

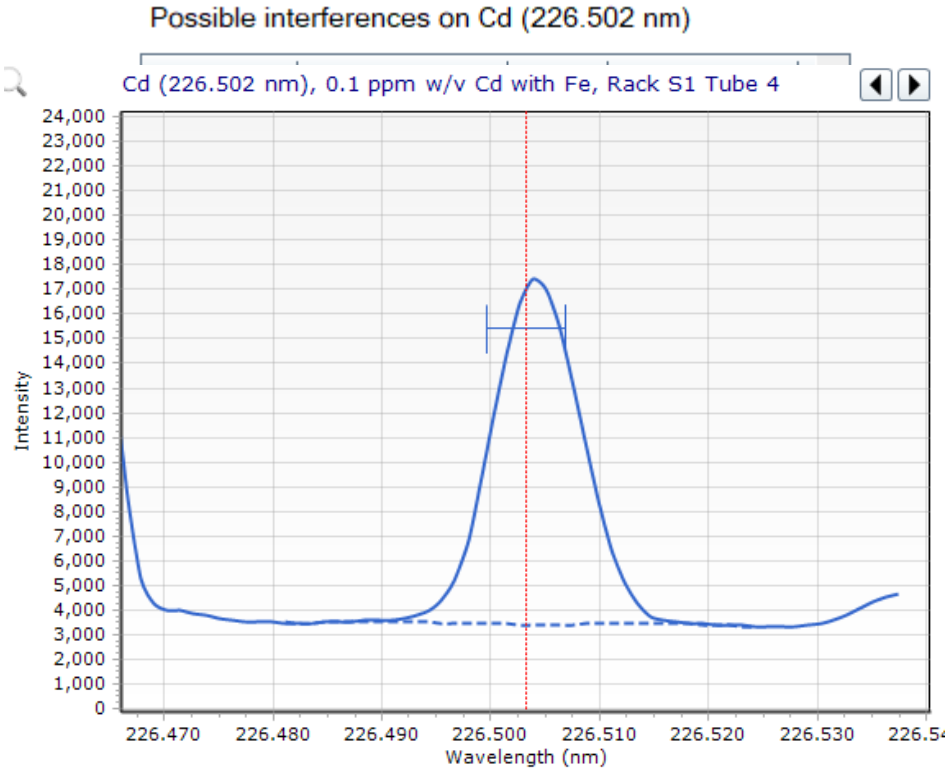
Fitted Background Correction (FBC)





- FBC calculates the true background signal – improving accuracy.
- Off Peak Left and Right background correction needs to be adjusted manually for the sample shown on the bottom left.
- FBC requires no user adjustment unlike Off Peak background correction.

Removing Spectral Interferences
FACT – Fast Automated Curve- Fitting Technique



- Resolves extremely complex spectral interferences.
- Allows resolution of interferences as close a 3 pm.
- Re-analysis of samples not required.
- Quick and Easy to set up.

Removing Spectral Interferences
FACT – Fast Automated Curve- Fitting Technique

Possible interferences on Cd (226.502 nm)

Symbol	Wavelength (nm)	Ion	Intensity
Ni	226.446	II	3752.8
Nb	226.455	II	122.8
Fe	226.459	II	16.0
Ir	226.461	I	98.0
Os	226.465	I	10.4
Ta	226.465	II	1.6
Ru	226.470	I	3.3
Mo	226.473	II	4.8
Co	226.488	I	10.6
Mo	226.495	II	2.5
Cd	226.502	II	38378.5
Fe	226.505	I	4.7
Ti	226.510	II	7.8
Mo	226.510	II	1.5
Ir	226.516	II	112.1
Cr	226.519	II	0.6
Nb	226.521	II	0.9
Co	226.526	II	2.1
W	226.533	II	78.4
Ni	226.535	II	12.5

- Resolves extremely complex spectral interferences.
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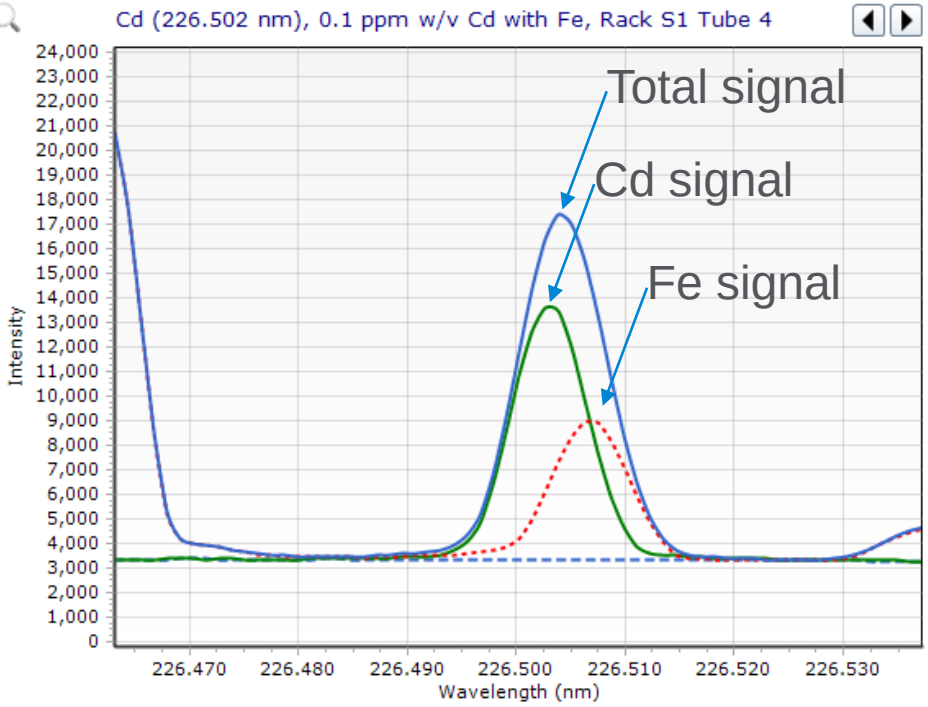
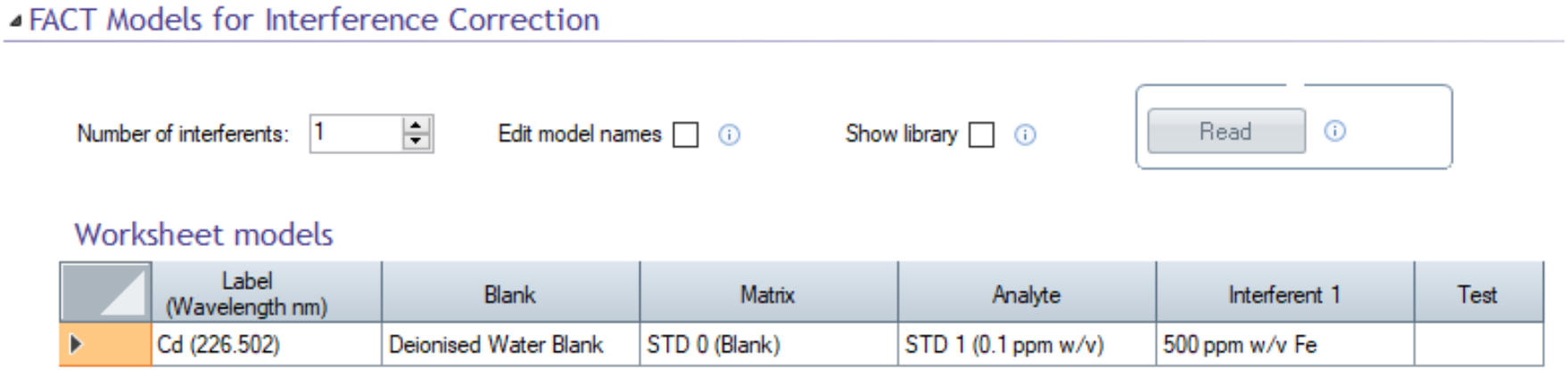
FACT – How Does it Work?



- Uses a peak modelling approach.
- Models the background, analyte and the interference.
- Deconvolutes the analyte peak from nearby interference peaks.
- When a model is created it can be stored in the library and used again.

FACT – In Practice



	Label (Wavelength nm)	Blank	Matrix	Analyte	Interferent 1	Test
▶	Cd (226.502)	Deionised Water Blank	STD 0 (Blank)	STD 1 (0.1 ppm w/v)	500 ppm w/v Fe	

- Select the solution to use as a blank.
- Select the solution to use as a matrix.
- Select the solution to use as the analyte.
- Select the solution to use as the interferent.

FACT – In Practice

Without FACT

Solution Label	Cd 214.439 nm ppm	Cd 226.502 nm ppm
1-Rcd 1 280514-1/1	0.095	0.122
2-14E029905-021/1	0.030	0.055
3-14E029872-011/1	0.010	0.047
4-14E029905-002/1	0.042	0.065
5-14E029790-002/1	0.009	0.030
6-14E029896-002/1	0.012	0.035
7-14E029102-006/1	0.010	0.033
8-14E029896-011/1	0.019	0.087
9-14E029488-002/1	0.011	0.035
10-14E029943-001/1	0.010	0.057
11-14E029896-012/1	0.027	0.105
12-14E030194-025/1	0.004	0.024
13-14E029605-005/1	0.026	0.119

- FACT model saved to the library for future use.
- IEC can still be used, however, this requires the interferents to be know before the analysis.
- In conjunction with IntelliQuant this can be a very powerful and quick spectral deconvolution tool.

FACT – In Practice

With FACT

Solution Label	Cd 214.439 nm ppm	Cd 226.502 nm ppm
1-Rcd 1 280514-1/1	0.089	0.089
2-14E029905-021/1	0.024	0.024
3-14E029872-011/1	0.001	0.001
4-14E029905-002/1	0.036	0.036
5-14E029790-002/1	0.004	0.004
6-14E029896-002/1	0.006	0.005
7-14E029102-006/1	0.004	0.004
8-14E029896-011/1	0.004	0.005
9-14E029488-002/1	0.005	0.004
10-14E029943-001/1	0.000	0.001
11-14E029896-012/1	0.008	0.008
12-14E030194-025/1	0.000	0.001
13-14E029605-005/1	0.005	0.004

- FACT model saved to the library for future use.
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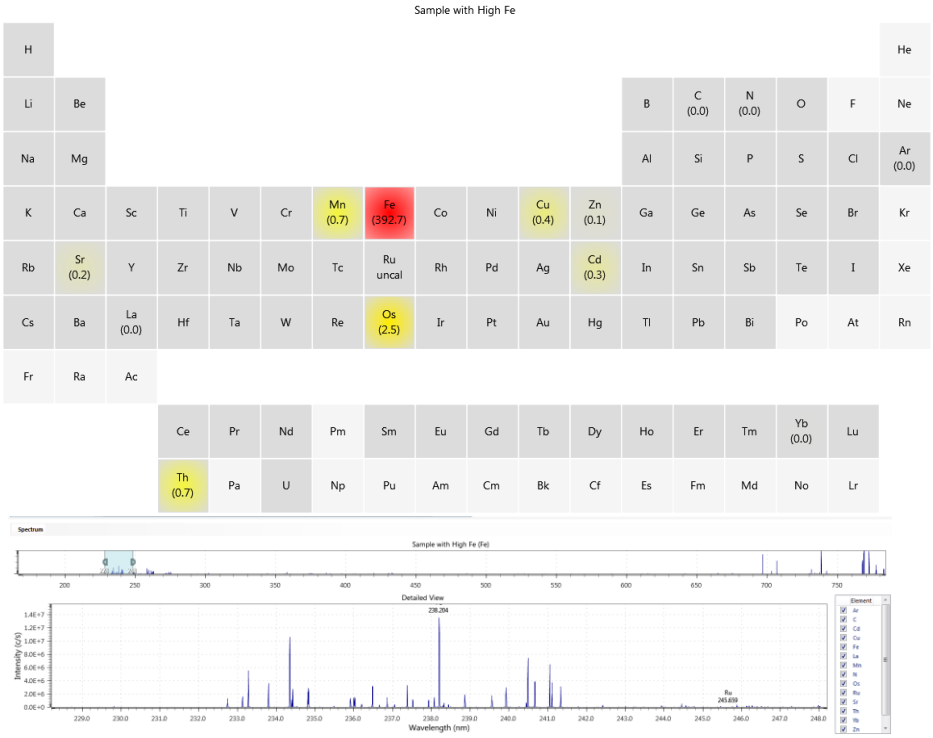
IntelliQuant



- Uses built in calibrations for semi-quantitative data.
- Up to 70 Elements.
- Only adds an additional 10-15 seconds to the analysis time.
- An Heat Map is generated showing elements present.

Summary

Vertical torch reduces blockages.
Argon humidifier to avoid neb clogging.

Dedicated sample introduction system (S/C, torch),
Control of the S/C temperature

Fitted and FACT background correction ensure the right results are reported.

IntelliQuant gives extra sample information – excellent for determining interferences.

The Agilent Atomic Spectroscopy Portfolio

Agilent's **55** and **200 Series** includes *Fast Sequential flame AA* and *high performance furnace.*

Agilent's **4210 MP-AES** runs on air for the *lowest cost of ownership* and *improved safety.*

Agilent's **5110 ICP-OES** includes the *world's most productive, and only Synchronous Vertical Dual View* ICP-OES.

Agilent's **7800** & **7900 ICP-MS** are *robust, sensitive, accurate, and easy to use quadrupole ICP-MS*

Agilent's **8900 ICP-QQQ with MS/MS mode** provides *unique control of interference removal in reaction mode*

Full portfolio to cover your elemental analysis needs

Tack!

QUESTIONS?

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