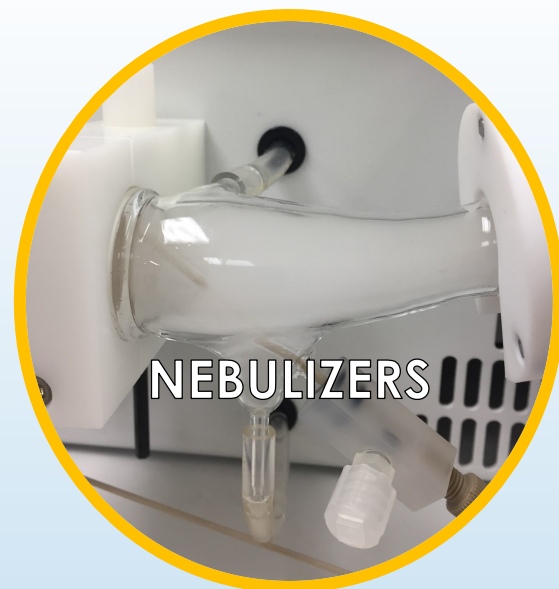
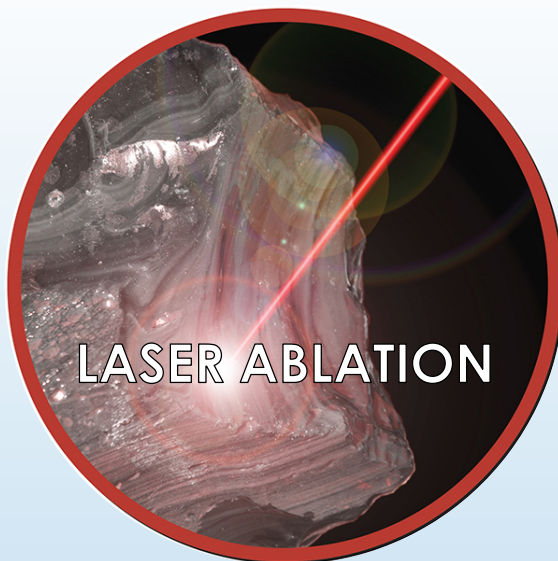




TELEDYNE CETAC TECHNOLOGIES
Everywhereyoulook™

Liquid Sample Introduction Techniques

***Measurement of Nutrient & Trace Level Elements
in Juices and Food Matrices Using Ultrasonic
Nebulization with ICP-OES Detection***



Introduction

Analysis of Juices and Foods

Consumers are more concerned about levels of potentially toxic elements such as As, Cd, and Pb in beverages and foods. Of heightened concern are food products intended for consumption by infants and children.

Reliable and precise methods of food analysis are important to maintain the safety of the food supply. In addition, accurate data is needed to comply with food labeling requirements for nutrients such as Ca, Fe, K, and Na.

Liquid Sample Introduction

Ultrasonic Nebulization

An ultrasonic nebulizer is an accessory for ICP-OES that enables higher sample transport efficiency (versus a standard pneumatic nebulizer) to the plasma. This benefit can be helpful for detection of more difficult elements such as As, Pb, Sb, Se, and Tl using ICP-OES detection.

This webinar describes the use of ultrasonic nebulization for improved ICP-OES detection of trace elements in juices and food matrices.

Ultrasonic Nebulization

Principle of Operation

In place of a regulated gas flow for generation of a liquid sample aerosol (pneumatic nebulization), liquid sample is pumped across a quartz plate with an underlying oscillating (piezoelectric) crystal.

The oscillations of the crystal will break up the liquid flow and cause formation of a sample aerosol. Ultrasonic nebulization is typically up to 10x more efficient (versus a conventional pneumatic nebulizer) for conversion of liquid sample into a useable aerosol.

Teledyne CETAC U5000AT⁺ Ultrasonic Nebulizer

Dimensions and Weight



Width: 35.6 cm (14.0 in)

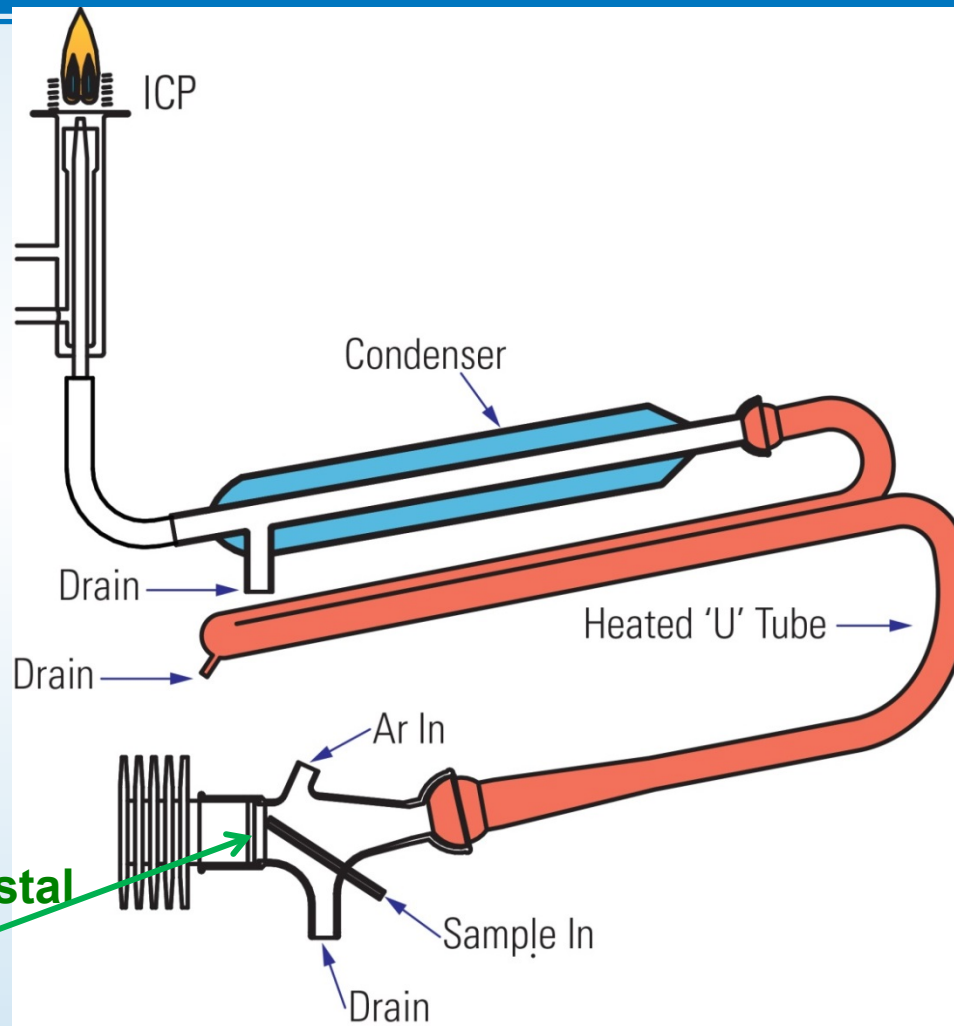
Depth: 34.9 cm (13.7 in)

Height: 25.4 cm (10.0 in)

Weight: 12.3 kg (27 lbs)

**Temperature
Controllers (2)**

Teledyne CETAC U5000AT⁺ Schematic



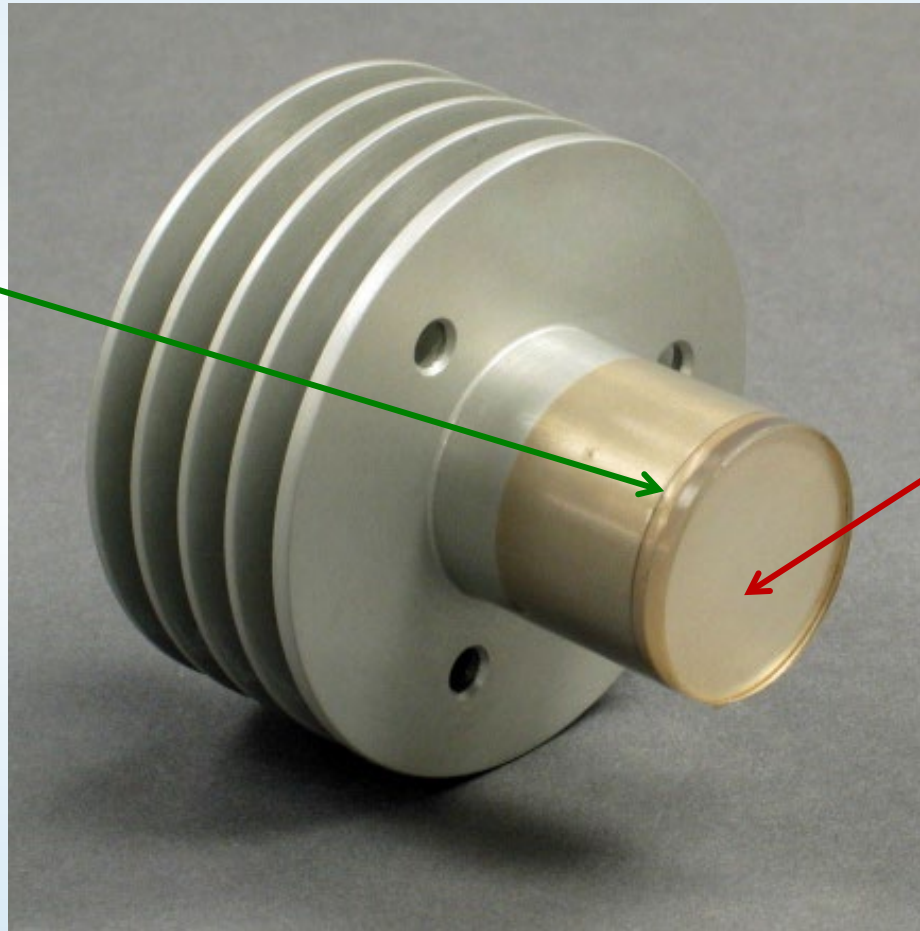
**Piezoelectric Crystal
(Transducer)**

U5000AT⁺ Important Features

- **Integrated desolvation system (electrothermal)**
 - Removes excess sample aerosol solvent for plasma stability
- **Built-in drain peristaltic pump**
 - Removes condensed sample solvent, no need to use host ICP-OES peristaltic pump

U5000AT⁺ Transducer Assembly

**Piezoelectric
Crystal
(disk shape)**



Quartz Plate

Instrumentation

ICP-OES: PerkinElmer Avio 500

Ultrasonic Nebulizer: Teledyne CETAC U5000AT⁺

Microwave Digestion System: CEM Mars 6

U5000AT⁺ Ultrasonic Nebulizer

Installation Steps

- 1. Remove standard nebulizer and spray chamber from the ICP-OES.**
- 2. Connect nebulizer gas inlet tubing from ICP-OES.**
- 3. Connect sample out tube from U5000AT⁺ to ICP torch.**
- 4. Connect power cord to U5000AT⁺ and turn on power. Allow 10 min. for Heater and Cooler to stabilize.**
- 5. Start ICP, introduce tune solution, press one button (Operate) to begin nebulizing tune solution and then samples. Adjust gas flows for best signal.**

U5000AT⁺ Ultrasonic Nebulizer

Installation Summary

Overall setup takes about 15 minutes.

Use host ICP-OES peristaltic pump to introduce samples.

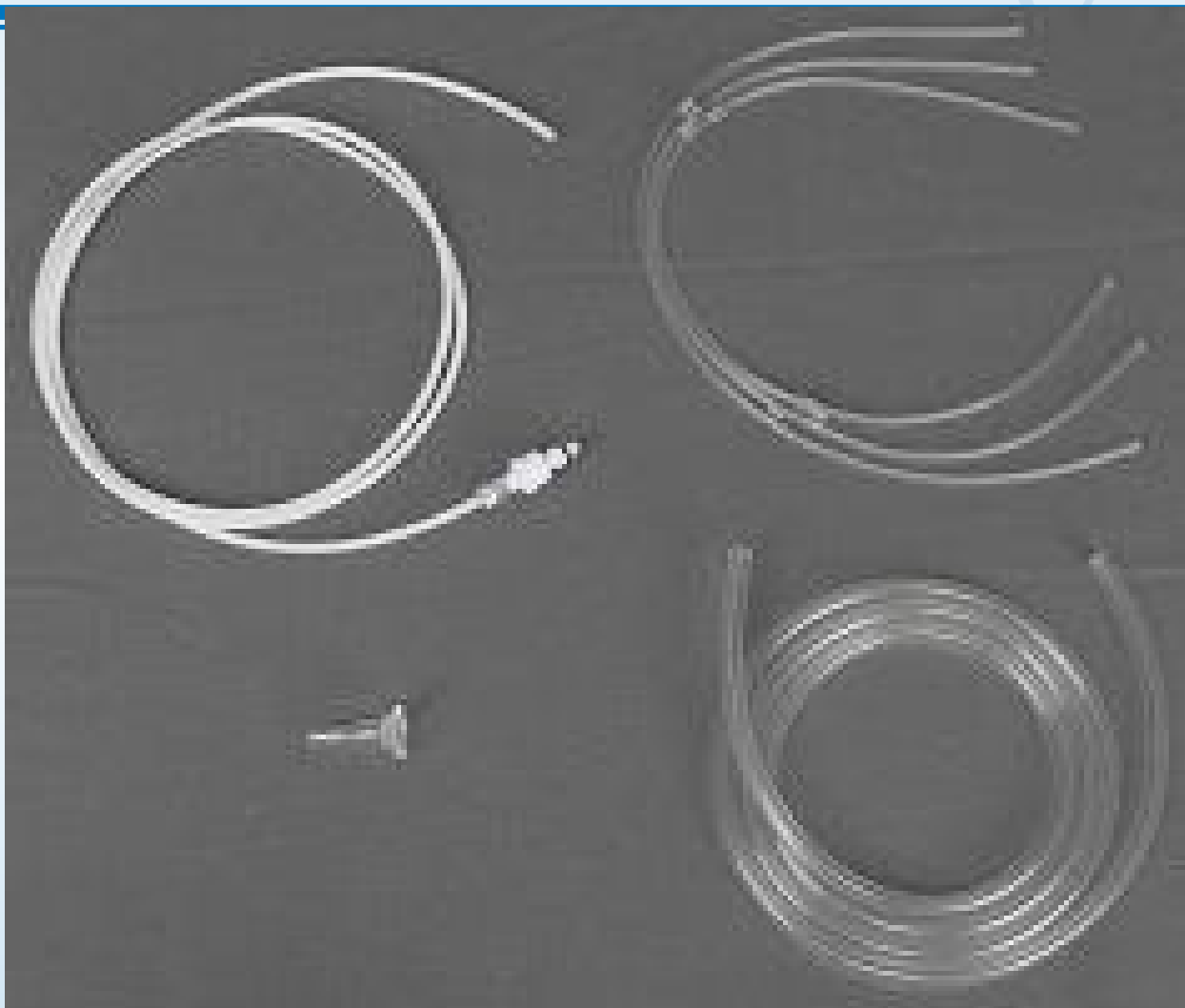
No computer control, no software installation needed.

One button operation

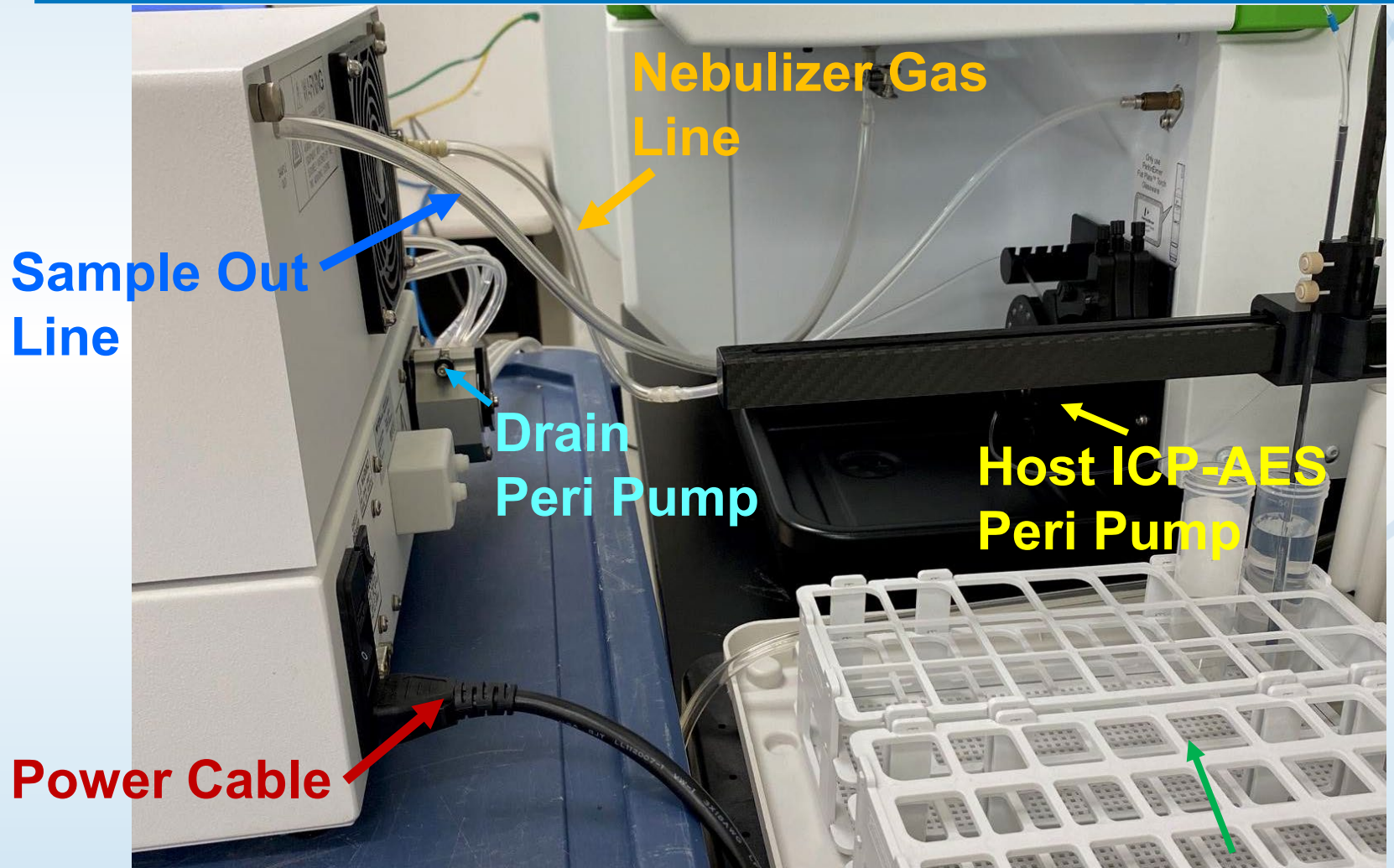
Sample introduction can be automated by connecting the sample inlet line to an autosampler (CETAC ASX-280 or ASX-560)

U5000AT⁺ Ultrasonic Nebulizer

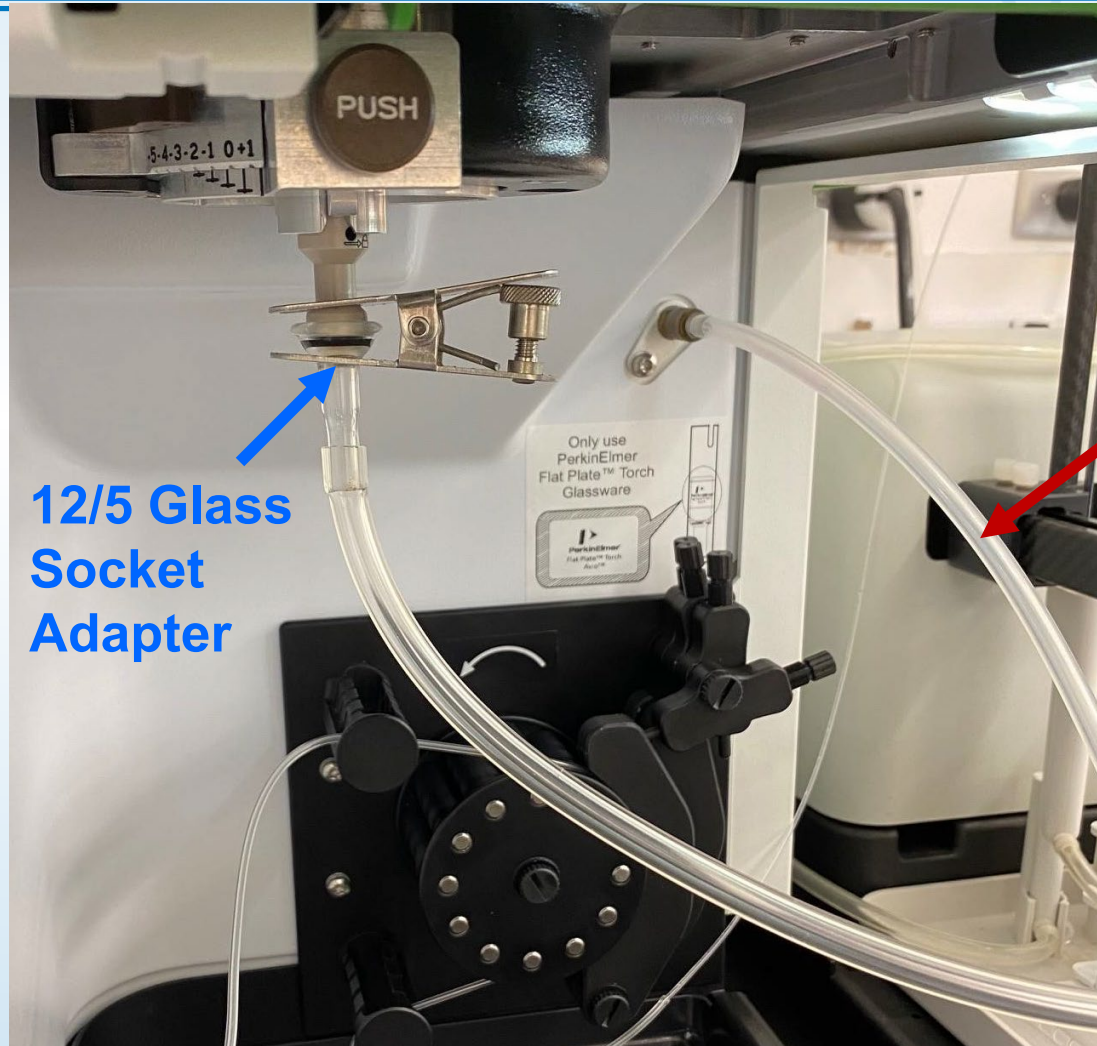
Example Interface Kit



Connections Between Host ICP-OES & U5000AT⁺ Ultrasonic Nebulizer



Torch Adapter Connection



12/5 Glass
Socket
Adapter

Neb Gas
Supply Line

ICP-OES with Ultrasonic Nebulization



PerkinElmer Avio 500 ICP-AES, U5000AT+ USN, ASX-280 Autosampler

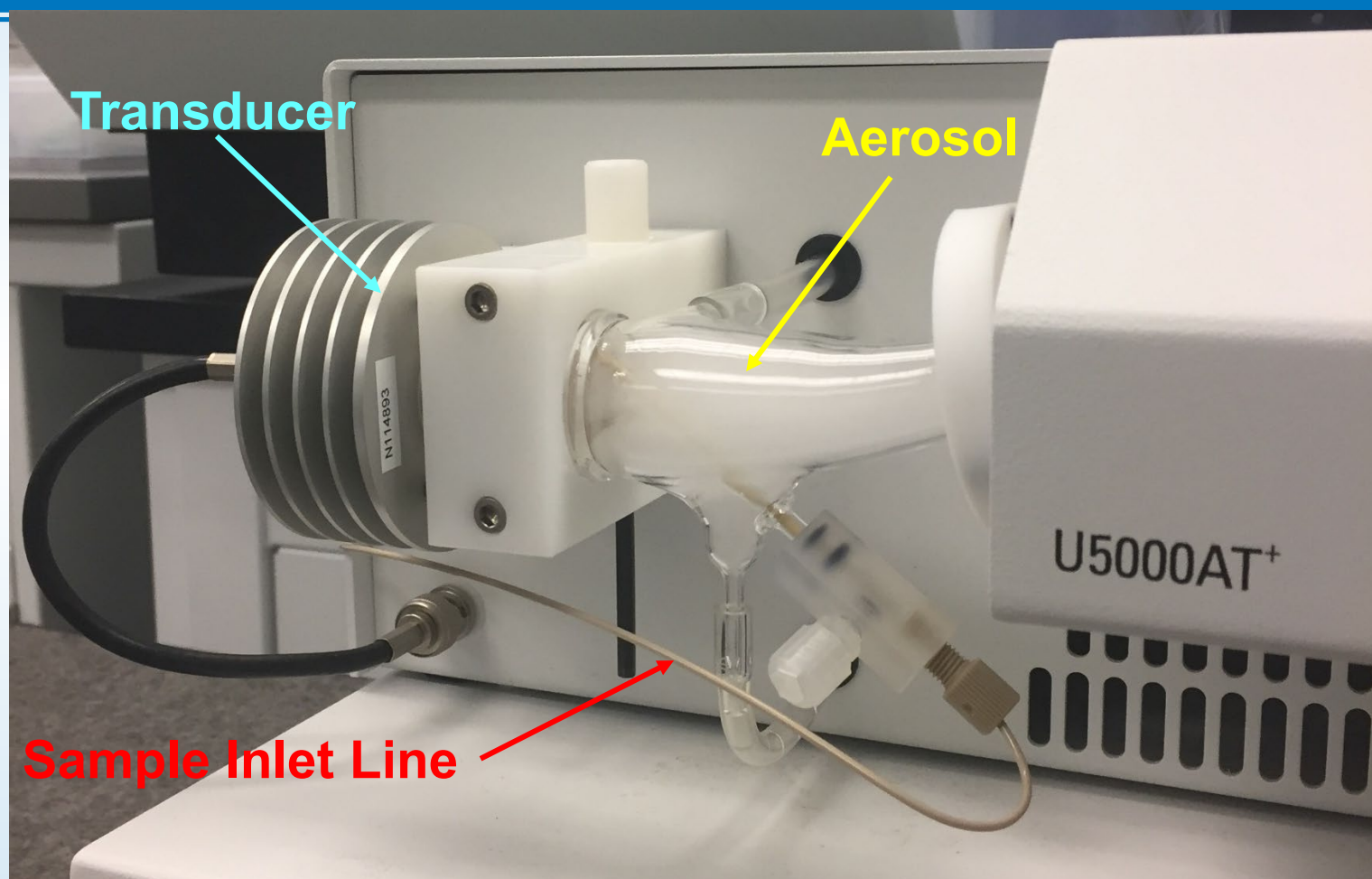
Ultrasonic Nebulizer

Aerosol Generation: One Button Operation



Ultrasonic Nebulizer Aerosol

Close-Up View



Operating Conditions

ICP-OES with Standard Nebulizer and U5000AT⁺ USN

Parameter	Std Nebulizer	U5000AT ⁺ USN
ICP Power	1500 W	1500 W
Plasma Gas	8.0 L/min	8.0 L/min
Auxiliary Gas	0.2 L/min	0.2 L/min
Nebulizer Gas	0.70 L/min	0.62 L/min
Torch Injector	2 mm	2 mm
Uptake Rate	1.0 mL/min	1.0 mL/min
Cassette Position	-3.0	-5.0
Resolution	Normal	Normal
Nebulizer Type	Meinhard K	Piezoelectric
Spray Chamber	Baffled cyclonic	Conical
Heater Temp	N/A	120°C
Cooler Temp	N/A	5°C
Integration Time	2 s min, 5 s max	2 s min, 5 s max
Peak Area	3 pts/peak	3 pts/peak
Replicates	3	3



Calibration

- The ICP-OES was calibrated using digested standards prepared in 10% HNO₃ and 4% H₂O₂
- Measure 1 blank and 3 standards, correlation coefficient must be better than 0.998
- Use on-line mixing tee (before nebulizer input) for addition of 100 µg/L Y internal standard

Note: H₂O₂ oxidizes As³⁺ to As⁵⁺ for improved As recoveries

Calibration Ranges and Wavelengths

Element	λ (nm)	View	Range
Ca	317.933	Radial	0.1-10 mg/L
Ca	315.887	Radial	1-100 mg/L
Mg	285.213	Radial	0.1-10mg/L
K	766.490	Radial	0.1-10 mg/L
Na	589.592	Radial	0.1-10 mg/L
Na	330.237	Radial	1-100 mg/L
Sb	206.836	Axial	10-1000 μ g/L
As	188.979	Axial	10-100 μ g/L
Ba	493.408	Radial	10-1000 μ g/L
Be	313.107	Axial	10-1000 μ g/L
Cd	214.440	Axial	10-1000 μ g/L

Calibration Ranges and Wavelengths

Element	λ (nm)	View	Range
Cr	205.560	Axial	10-1000 μ g/L
Cu	327.393	Axial	10-100 μ g/L
Fe	238.204	Axial	50-1000 μ g/L
Pb	220.353	Axial	10-1000 μ g/L
Mn	257.610	Axial	50-1000 μ g/L
Mo	202.031	Axial	10-1000 μ g/L
Ni	231.604	Axial	10-1000 μ g/L
P	178.221	Axial	10-1000 μ g/L
P	214.914	Radial	1-100mg/L
Se	196.026	Axial	10-1000 μ g/L
U	409.014	Axial	50-1000 μ g/L
Zn	206.200	Axial	50-1000 μ g/L

Sample Types

- **Beverages**
 - **100% Cranberry Juice**
 - **Apple Juice from a juice box**
 - **100% Fruit Punch from a juice box**
- **Foods**
 - **Dry Cereal**
 - **Baby Food from a pouch**
 - **Donut**
- **Standard Reference Materials**
 - **NIST® 1538 Rice Flour**
 - **NIST® 1577 Bovine Liver**
 - **NIST® 1549 Non-Fat Milk Powder**

Sample Weights and Volumes for Digestion

Sample Type	Amount Digested
NIST Standards	0.5 g
Dry Food (cereal)	0.5 g
Wet Food (baby food)	1 g
Juice	5 mL

Sample Preparation

1. To each digestion vessel add 8 mL conc. HNO_3 and 1 mL of 30% H_2O_2 .
2. Allow samples to react for at least 10 minutes in a ventilation hood.
3. Place cap on digestion vessels.

Microwave System Operating Parameters

Power	Ramp	Hold	Temp
1200 W	25 min	15 min	200°C

After digestions were complete, samples were cooled to room temperature and diluted to a final volume of 25 mL with deionized water.

Instrument Detection Limits

Ten reagent blanks (10% HNO_3 / 4% H_2O_2) were measured using the standard nebulizer kit and the U5000AT⁺.

Instrument detection limits (IDLs) are calculated as 10x the standard deviation of the blank concentration.

Instrument Detection Limits

Selected Elements

Element	Std Neb IDL ($\mu\text{g/L}$)	U5000AT ⁺ IDL ($\mu\text{g/L}$)	Improvement Factor
As	5.53	0.35	15.8
Cd	0.54	0.10	5.4
Pb	4.97	0.53	9.5
Se	7.62	0.97	7.8

Sample Spikes

Samples were spiked at concentrations of 10 µg/L and 50 µg/L for trace elements and 0.5 mg/L for Ca, Na, Mg, and K before digestion. Per the FDA EAM (Elemental Analysis Manual) 4.4, % spike recovery must be 80% -120% to pass.

If the analyte concentration was > 30% of the spike value, results are not reported.

- Juices

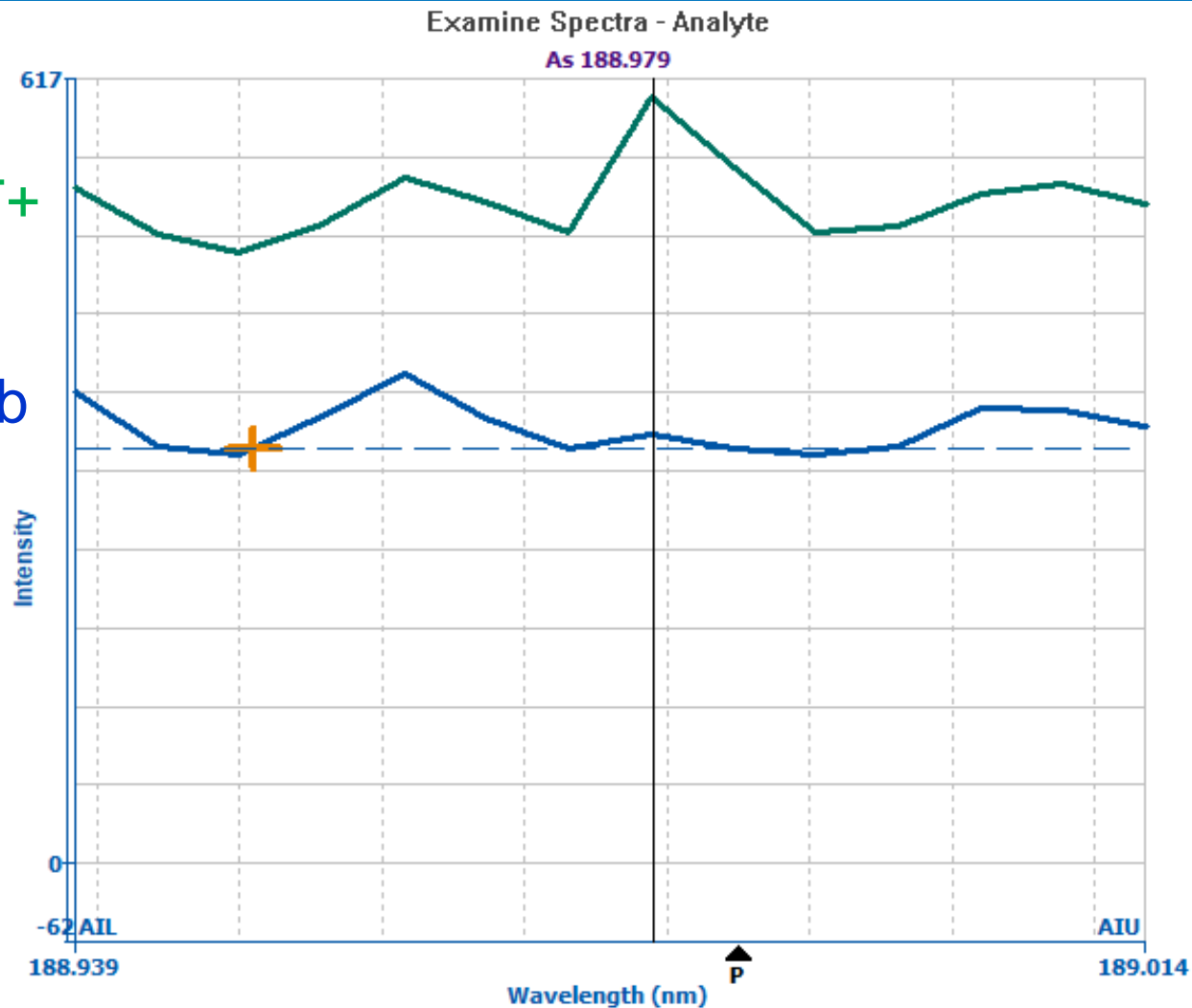
- Juices

Comparative Emission Spectra

10 $\mu\text{g/L}$ As spike in Cranberry Juice

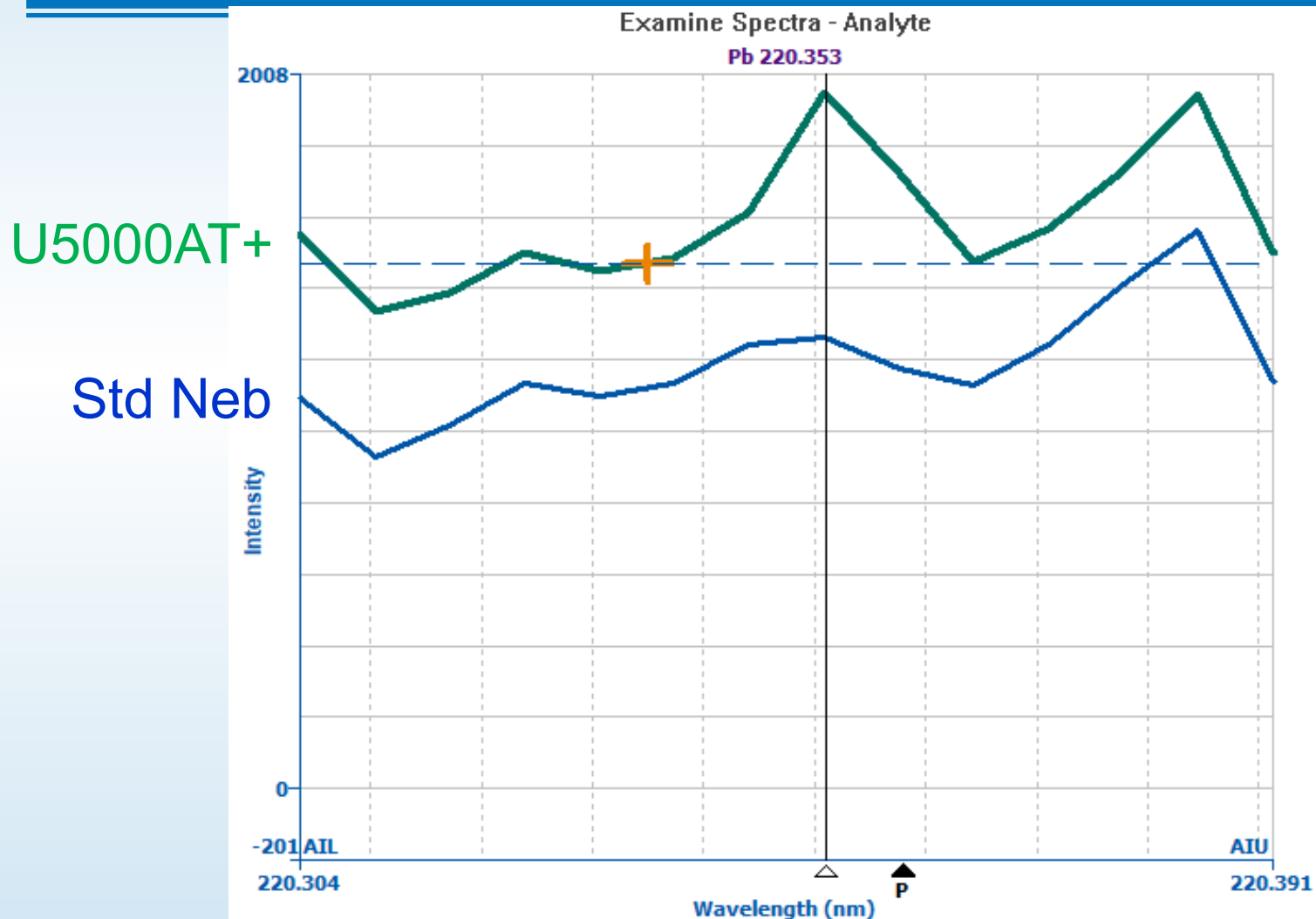
U5000AT+

Std Neb



Comparative Emission Spectra

10 $\mu\text{g/L}$ Pb spike in Cranberry Juice



Baby Foods

- **Type 1**
 - Blend of apples, carrots, blueberries and yogurt
- **Type 2**
 - Blend of bananas, blueberries and oats

Per Cent Spike Recoveries – Food Matrices

50 µg/L spike

Element	Baby Food Type1	Baby Food Type 2
As	94	108
Ba	88	95
Be	97	103
Cd	98	109
Cr	88	95
Cu	98	98
Mn	99	90
Mo	95	98
Ni	100	108
Pb	91	98
Sb	96	94
Se	77	99
U	86	84
Zn	103	117

Per Cent Spike Recoveries – Food Matrices

50 µg/L spike

Element	Glazed Donut	Breakfast Cereal
As	-	-
Ba	106	101
Be	111	99
Cd	111	108
Cr	104	104
Cu	111	101
Mn	-	110
Mo	110	107
Ni	104	100
Pb	97	97
Sb	95	99
Se	105	91
U	88	86
Zn	-	-

Standard Reference Materials

Standards were diluted at a minimum of 50x after digestion.

Elements with reference values below the detection limit were not reported.

Per FDA EAM 4.41, reference values should have a per cent recovery of 80% – 120%.

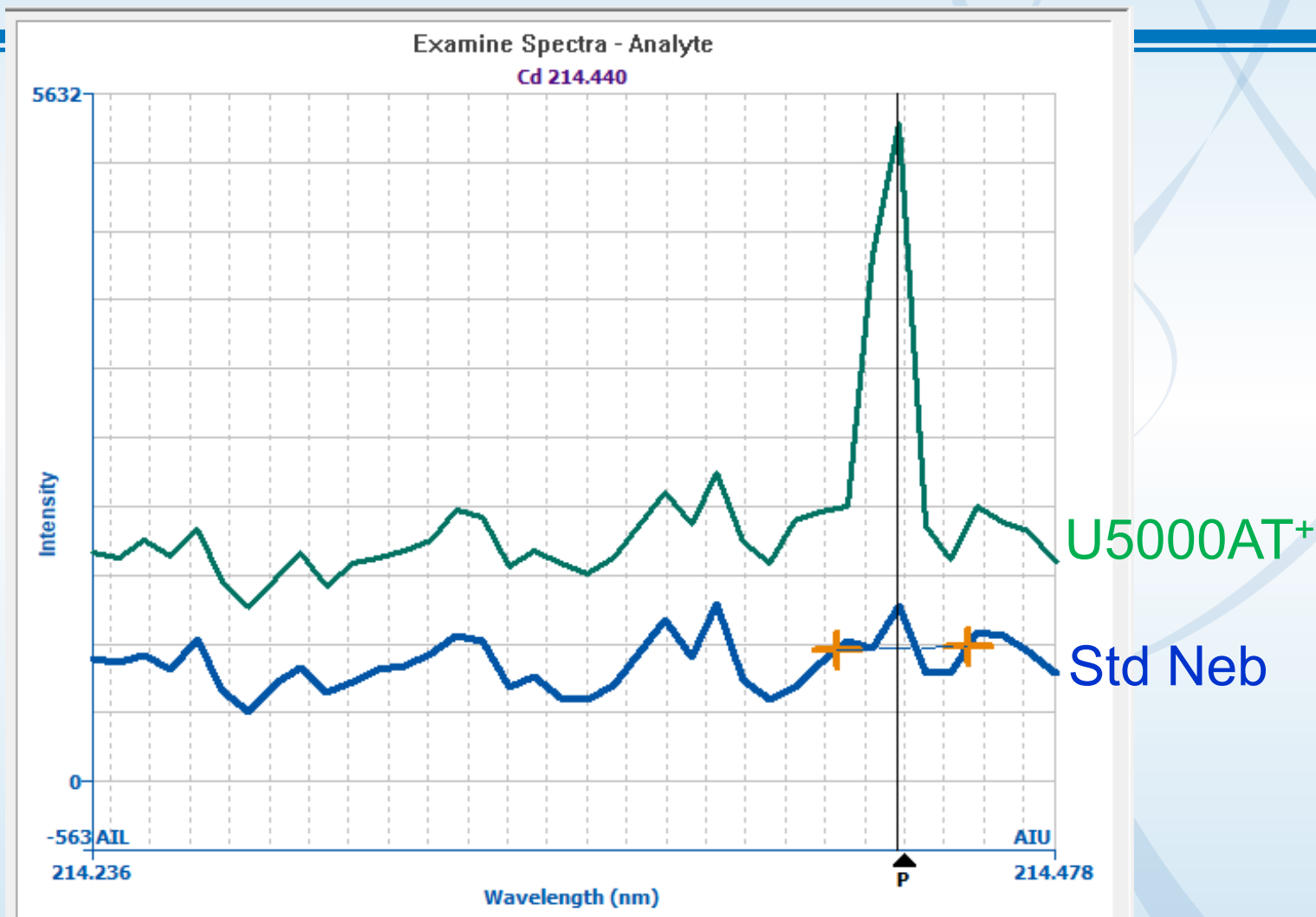
Certified Reference Materials

NIST® 1577 Bovine Liver

Element	Ref. Value (mg/kg)	Result (mg/kg)	% Recovery
Ba	-	-	-
Ca	120	116	97
Cd	0.44	0.497	113
Cu	158	148	94
Fe	194	186	96
K	9960	9410	93
Mg	600	547	91
Mn	9.9	9.4	95
Mo	3.5	3.37	96
Na	2430	2100	86
P	11100	9477	85
Zn	123	107	87

Comparison of Cd ICP-OES Signal

NIST® 1577 Bovine Liver



8.8 $\mu\text{g/L}$ Cd before dilution factor

Certified Reference Materials

NIST® 1538 Rice Flour

Element	Ref. Value (mg/kg)	Result (mg/kg)	% Recovery
Ba	-	-	-
Ca	118	112	95
Cd	-	-	-
Cu	2.4	2.42	101
Fe	7.4	7.6	102
K	1280	1189	93
Mg	560	513	92
Mn	20	19	95
Mo	1.46	1.43	98
Na	-	-	-
P	1530	1732	113
Zn	19.4	19.3	99

Certified Reference Materials

NIST® 1549 Nonfat Milk Powder

Element	Ref. Value (mg/kg)	Result (mg/kg)	% Recovery
Ba	2.2	2.06	94
Ca	13000	12245	94
Cd	-	-	-
Cu	-	-	-
Fe	1.78	1.68	95
K	16900	14694	87
Mg	1200	1186	99
Mn	-	-	-
Mo	-	-	-
Na	4970	4679	94
P	10600	14694	87
Zn	46.1	54.5	118

Analysis of Juices and Food Matrices Using U5000AT⁺ & ICP-OES Detection:

Conclusions and Benefits

- **Enhanced signal and lower detection limits for important elements such as As, Cd, Pb, and Se**
- **Accurate nutrient results necessary for food package labels**
- **Fast and easy setup with host ICP-OES**
- **No computer control required**

Ultrasonic Nebulizer for ICP-OES

Part Numbers for Ordering

- **U5000AT⁺ Ultrasonic Nebulizer**
 - **U51-99-0001A⁺ (115 V)**
 - **U51-99-0001B⁺ (220 V)**

U5000AT⁺ Interface Kits for ICP-OES

U5000AT⁺ Compatibility

- **All ICP-OES Models**
 - **Examples with Interface Kit SP Numbers ()**
 - **Agilent (SP5155Y)**
 - **Analytik Jena (SP5155Y)**
 - **PerkinElmer (SP5155L)**
 - **Shimadzu (SP5155Z)**
 - **Spectro (SP5155P)**
 - **Teledyne Leeman (SP5155I)**
 - **Thermo Fisher (SP5155CC)**

One interface kit provided at no charge with each U5000AT⁺

Acknowledgement

- **Special thanks to Mary Jo Menke-Wright for her work in sample preparation, method development, data collection, and outline text for this important application.**

Where to go for more information

- Service support
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- Technical support
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 - Paula.Doeschot@Teledyne.com
- Web information
 - <https://www.teledynecetac.com/products/nebulizers/u5000at+>
 - https://www.teledynecetac.com/site-products/Brochures/Flyer_U5000AT.pdf
 - <https://www.teledynecetac.com/resourceSite/Application%20Notes/AP-U5000-005.pdf>