Inductively Coupled Plasma – Optical Emission Spectrometer

Last updated 20230215

CORAL

You must be a "Qualified Self-User" to operate this instrument
When you arrive, Engage the instrument using CORAL.
When you leave, Disengage the instrument using CORAL.
Any problems, STOP, Post a note on the instrument and send an email to mtim@mit.edu immediately.

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Room Hazards (13-4139 & 13-4148)

Cryogenic Liquids, Chemicals & Lasers.

ICP Safety

Required Apparel – Gloves, Lab Jacket, Safety Glasses, Goggles
No Hydro Fluoric Acid usage.
No Viable Biological Samples.

EMERGENCIES DIAL100 (From an MIT phone)
MIT Police (617) 253-1212

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Agilent 5100 VDV ICP-OES

https://mtim.mit.edu/icp-oes

Massachusetts Institute of Technology
Materials Research Laboratory
Materials Research Shared Engineering Center
Analysis Shared Experimental Facility
Optical, Thermal Characterization Laboratory

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Checkout the system when you arrive.

Exhaust Hood Air Flow – Grater than 150SCFM.
Liquid Argon Dewar –95psi delivery pressure, Regulator Valve - Closed.
Waste container – Three drain lines. Good condition, not full, red tag.
Chiller – Off.
Tubing – Loose.
Computer – On.
Software – Closed.
Autosampler – Green Light.
ICP – Yellow Flashing Light.

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Startup

Liquid Argon Regulator Valve – Open
Chiller – On.
Tubing – Tightened.
Rinse Solution – Verify there is enough for your work. Spare is kept in the hood.
Logon to the computer – Username: Agilent, Password (Blank).
Open Software – "ICP Expert".
(Gas Module Error?
In the Software on the Instrument Status window select the Polychromater Boost momentarily).
Light the plasma – Wait 20 minutes before collecting data.
Autosampler – Put probe in rinse solution.
Put your samples & standards in the autosampler.
Create or open a measurement file.
(Note: if looking for any lines below 190nm turn on the snout purge on the instrument status panel).

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Before running the measurement, Verify:

No Leaks.
Periodic Bubbles in the Spray Chamber Drain Line.
Periodic Bubbles in the Autosampler Drain Line.
Stable fog in the sample chamber.
Stable torch flame.

Run the measurement

Measurement End:
Create a data report.
Export files as CSV if needed.
Walkaway with a copy of your data – No data storage here.
Do not count on your data being stored on this computer.

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Shutdown

Turn Plasma Off
Dry the tubing:
    Raise the autosampler probe
    Turn sample pump on.
Close the Liquid Argon Regulator Valve.
Wait 10 minutes:
Turn the pump - Off.
Software - Closed.
Chiller - Off.
Pump Tubing - Loose.
Hood Sash – Down.
Cleanup – Do not leave your samples here.
Close the Liquid Argon Regulator Valve Only.

Finishing Up

Disengage CORAL
Report any problems, comments or suggestions to: mtim@mit.edu

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Problems

It is ok to restart the computer and Spectrometer. If the problem persists, stop work and email mtim@mit.edu immediately.

Emergency Shutdown

Ok to leave as is.
Secure your chemicals.
Walk away.

Exhaust Hood Alarm

Call FIXIT.

Startup after a power loss

Exhaust hood air must be flowing – If needed, call FIXIT.
Restart Spectrometer Power – Switch on side.
Restart Autosampler Power – Switch on back.
Restart Computer.

Procedure for Extended Shutdown

Secure chemicals in exhaust hood.
Turn Off Spectrometer Power – Switch on side.
Turn Off Autosampler Power – Switch on back.
Turn Off Computer.
Purge the spectrometer.

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All samples and standards must be filtered with a 0.2um filter.

Fisher Scientific 25mm, 0.2um. PES, F Leur Lock Syringe Filter
https://www.fishersci.com/shop/products/choice-pes-polyethersulfone-syringe-filters/03253890#

Disposable Syringe:
https://www.coleparmer.com/i/cole-parmer-clear-disposable-syringe-luer-lock-tip-syringe-non-sterile-30-ml-50-bag/0794540

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Agilent 5100 VDV

Radial or Axial viewing of the plasma
Autosampler SP4
HF Introduction System
ICP Expert Data Analysis Software
Measurement Range: 167nm - 785nm
S/N: MY15470004
MRL CORAL Name: ICP_AES_5100

Wavelength Calibration Solutions	Part Number
ICP-OES wavelength calibration concentrate, 500 mL Contains 50 mg/L Al, As, Ba, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sr, Zn and 500 mg/L K in 5% HNO_3 (Dilute 10x before use)	6610030000
ICP-OES wavelength calibration solution, 500 mL Contains 5 mg/L Al, As, Ba, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sr, Zn and 50 mg/L K in 5% HNO_3 (Ready to use)	6610030100
ICP-OES calibration blank solution, 500 mL Pure ASTM Type 1A water with 5% HNO_3 (For use as a calibration blank or diluent for the wavelength calibration concentrate)	5190-7001

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Vendor Contacts

Inorganic Ventures: Standards and sample prep recipes: Amber Compton, a.compton@inorganicventures.com (540) 585-3030

Agilent Applications Specialist: Christopher Conklin, Christopher.conklin@agilent.com

Milestone (Sample prep and digestion company): Stephen White, s.white@milestone.com

Other Instruments on Campus

MIT Lab for Nuclear Science – ICP-OES.

MIT CEHS: ICP-MS Microwave Digester, (New Contact),
https://cehs.mit.edu/core-facilities/bioimaging-and-chemical-analysis-facilities-core

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Applications

- Agricultural and food
  - Animal tissues, beverages, feeds, fertilizers, garlic, nutrients, pesticides, plant materials, rice flour, soils, vegetables, wheat flour
- Biological and clinical
  - Brain tissue, blood, bone, bovine liver, feces, fishes, milk powder, orchard leaves, pharmaceuticals, pollen, serum, urine
- Geological
  - Coal, minerals, fossils, fossil fuel, ore, rocks, sediments, soils, water
- Environmental and water
  - Brines, coal fly ash, drinking water, dust, mineral water, municipal wastewater, plating bath, sewage sludge, slags, seawater, soil
- Metals
  - Alloys, aluminum, high-purity metals, iron, precious metals, solders, steel, tin
- Organic
  - Adhesives, amino acids, antifreeze, combustion materials, cosmetics, cotton cellulose, dried wood, dyes, elastomers, epoxy, lubricant, organometallic, organophosphates, oils, organic solvent, polymers, sugars
- Other materials
  - Acids, carbon, catalytic materials, electronics, fiber, film, packaging materials, paints and coatings, phosphates, semiconductors, superconducting materials

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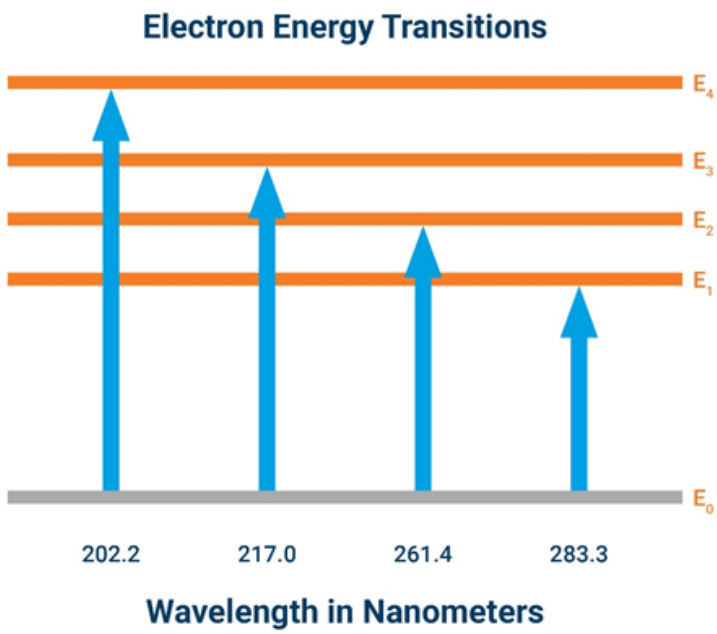
Theory

- ICP-OES is an analytical technique that is used to determine how much of certain elements are in a sample.
- As atoms and ions absorb energy in a plasma the electrons move from ground to an excited state.
- As the electron returns it emits light of a specific wavelength.
- The type of atom or ion and the energy level transition determines the wavelength of the emitted light.

Lead (Pb) Emission Lines



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Quantitative

- The amount of light released at each wavelength is proportional to the number of atoms or ions making the transition.
- The Beer Lambert Law is used to describes the relationship between light intensity and concentration of the element.

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Main Measurement Steps

- Select the elements in the sample to be measured.
- Prepare solutions of the sample using conventional techniques of quantitative chemical analysis.
- A set of calibrating solutions is prepared. Solutions containing known concentrations of the analyte elements. The range of concentrations for each element must encompasses the expected concentration of that element in the sample solutions (if known).
- Deliver the calibration solutions and sample to the plasma and measure the intensity of light at each wavelength.
- Calibration graphs are prepared for each element from the emission intensities of the calibrating solutions.
- The concentrations of the elements in each sample solution are determined from the calibration graphs, typically in µg/L or mg/L.

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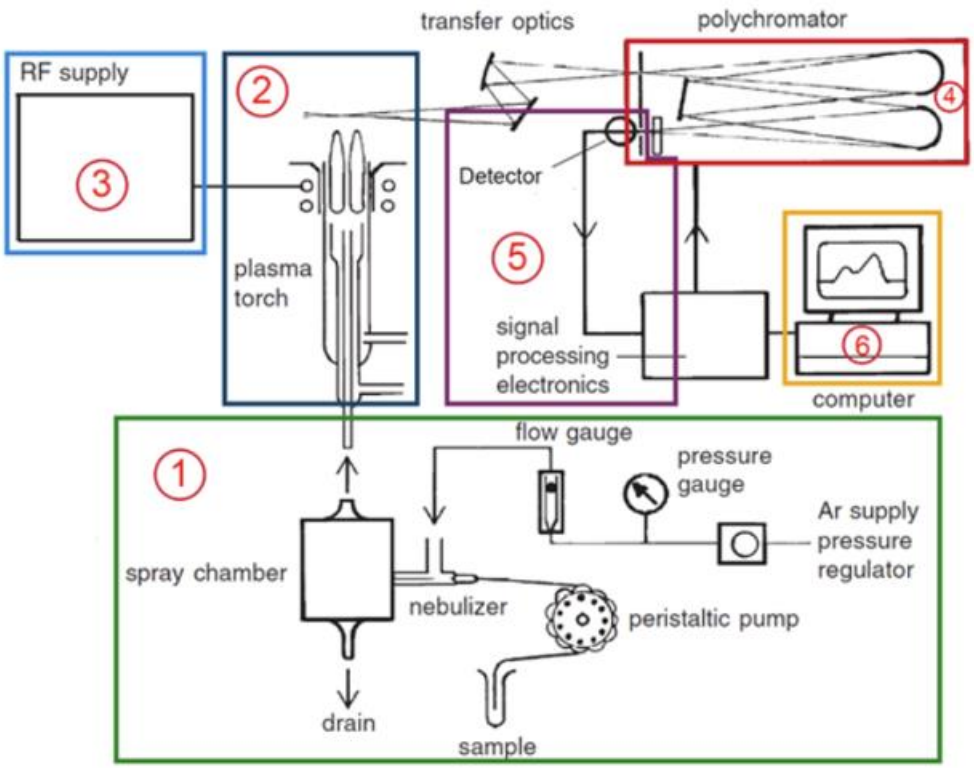

Main Components

1. *A sample introduction system*
2. *The plasma torch and its associated gas supplies*
3. *A radio-frequency generator*
4. *An optical spectrometer*
5. *Detectors and associated electronics*
6. *Computerized instrument control, data collection, and analysis.*

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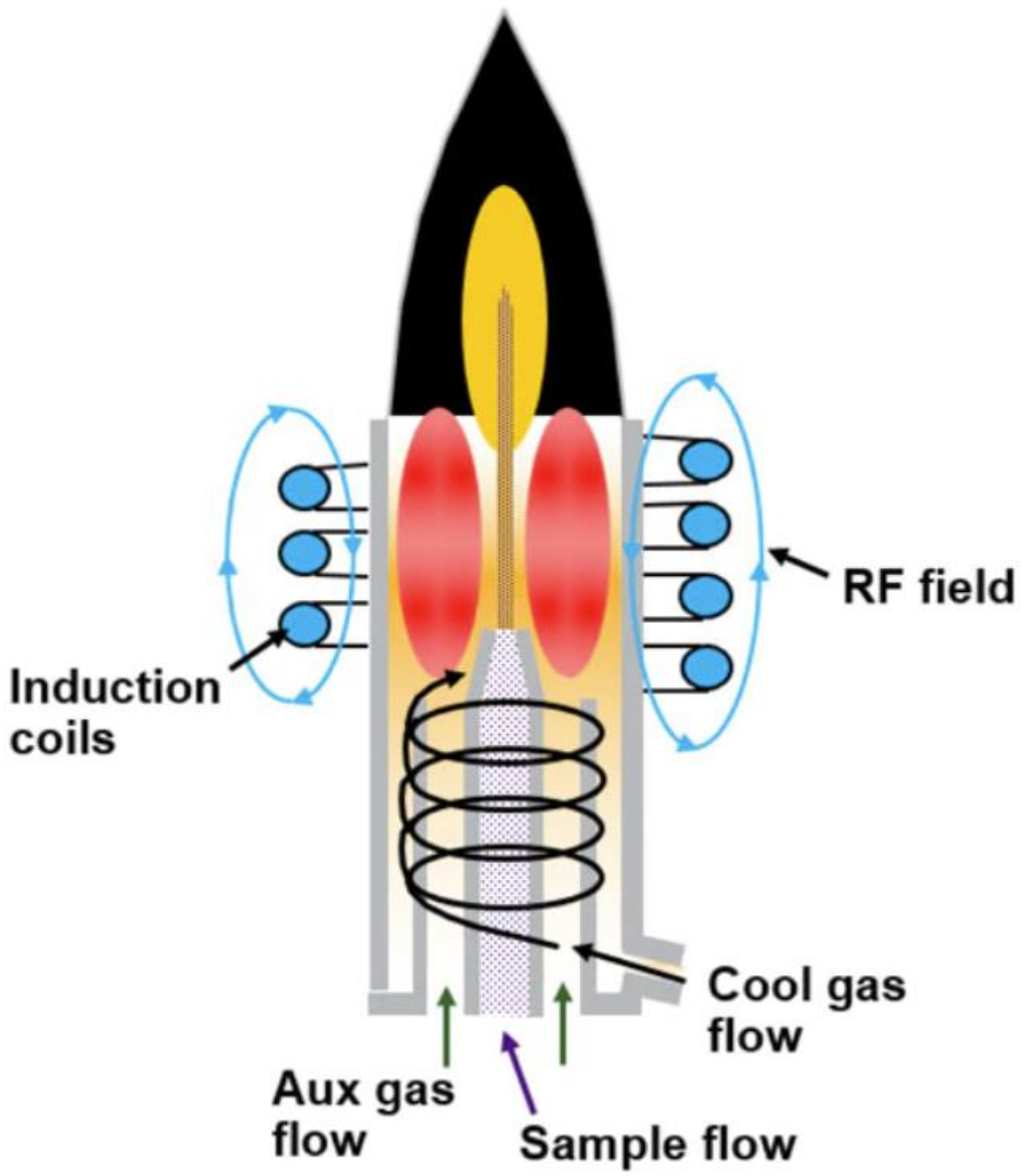

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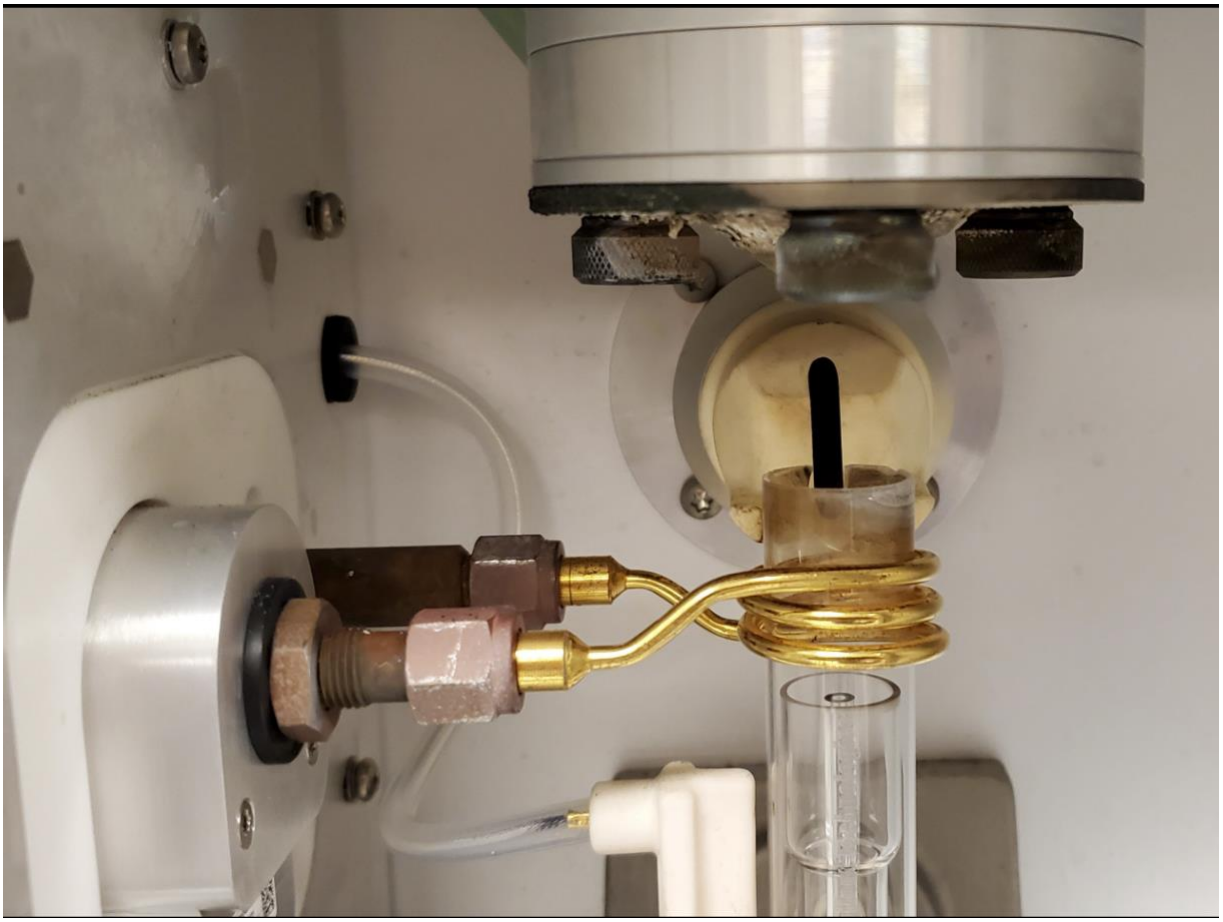

Dual View

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Radial

- High Precision
- Detection Limits - high ppb or ppm
- Reduced background emission and matrix interference
- High salts
- High totally dissolved solids
- Wear metals in oils
- Organic solutions

Axial

- High Sensitivity
- Detection limits 2-3x lower than radial – ppb
- High background and matrix effects
- Trace metals in water (environmental)
- • Trace metals in any kind of material (for chemical, metal, or pharmaceutical industries, etc.)
- • Trace metals in wastewater and other effluents

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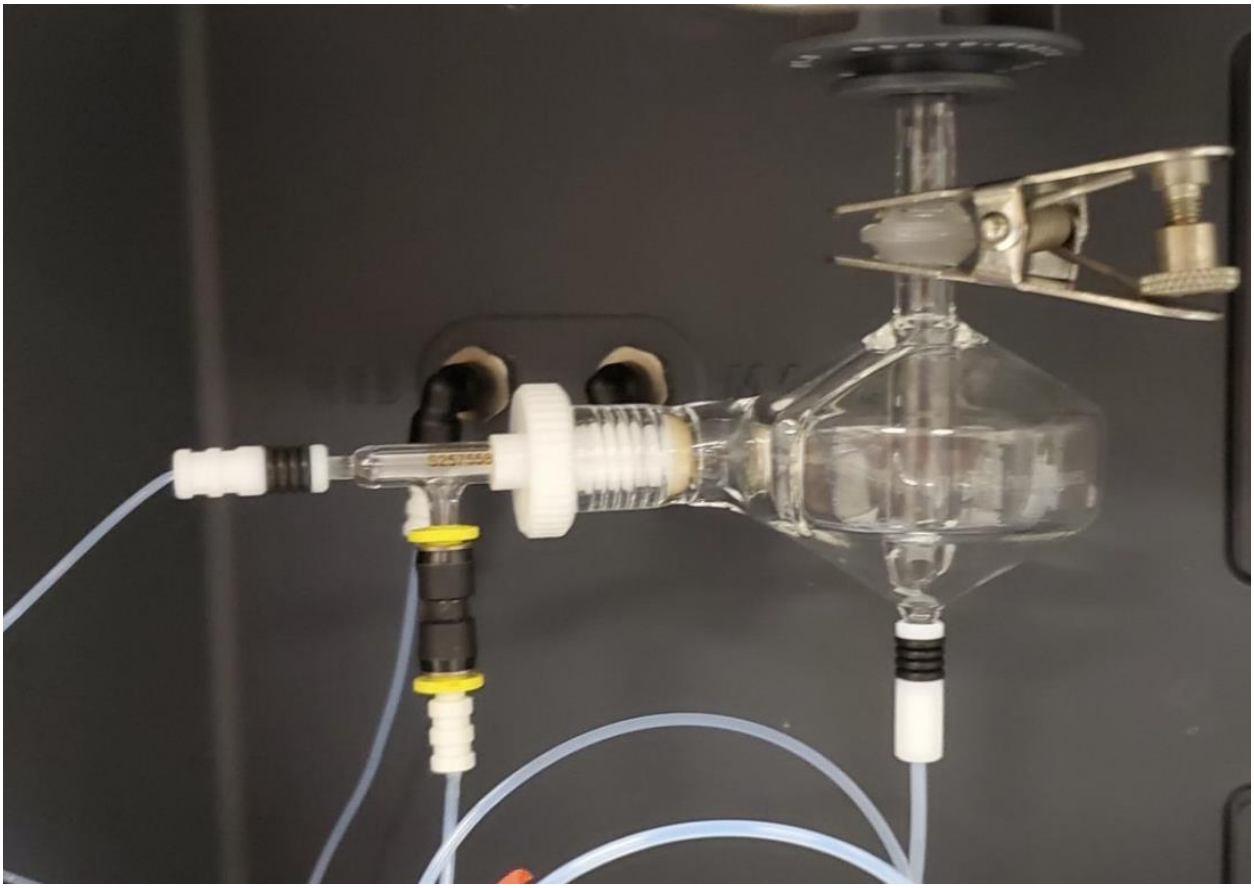

Nebulizer & Spray Chamber

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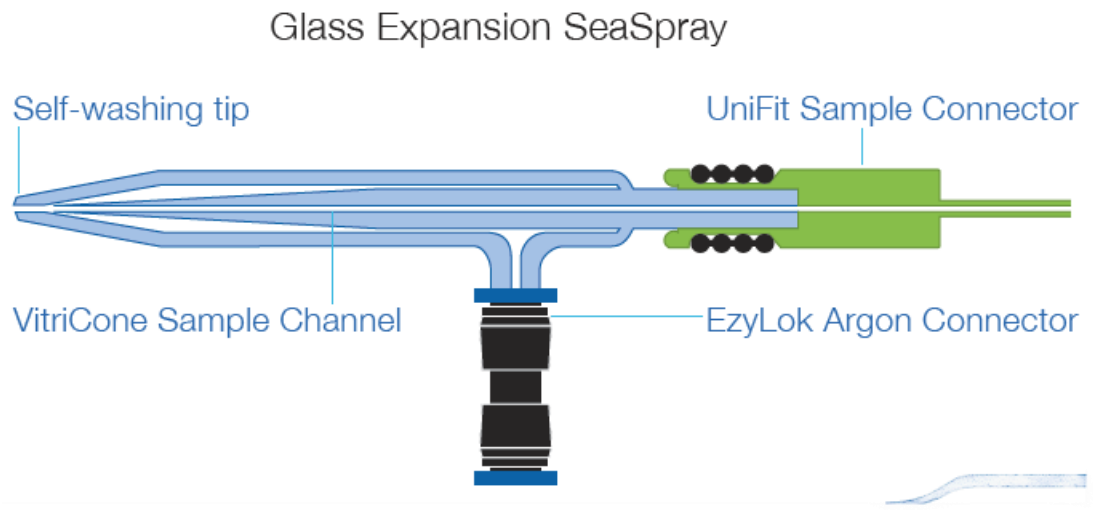

Nebulizer

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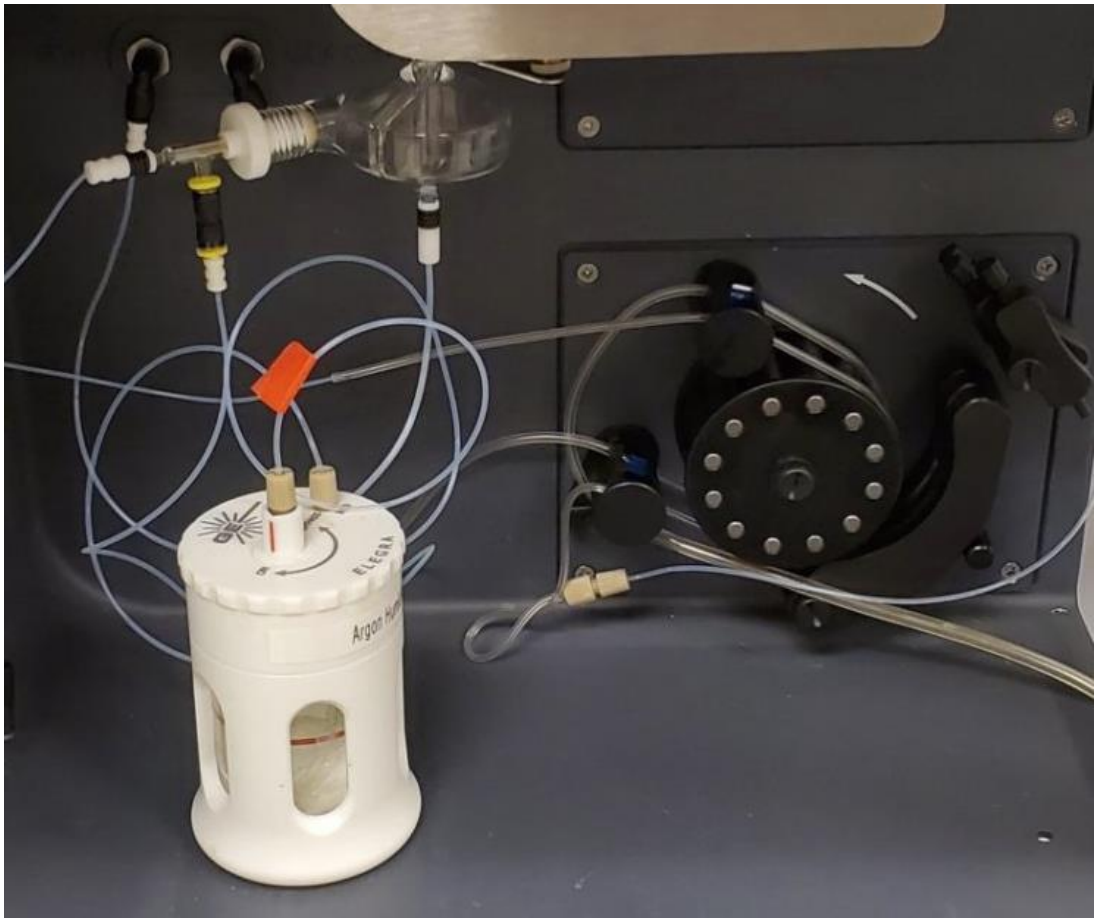

Sample Introduction Area

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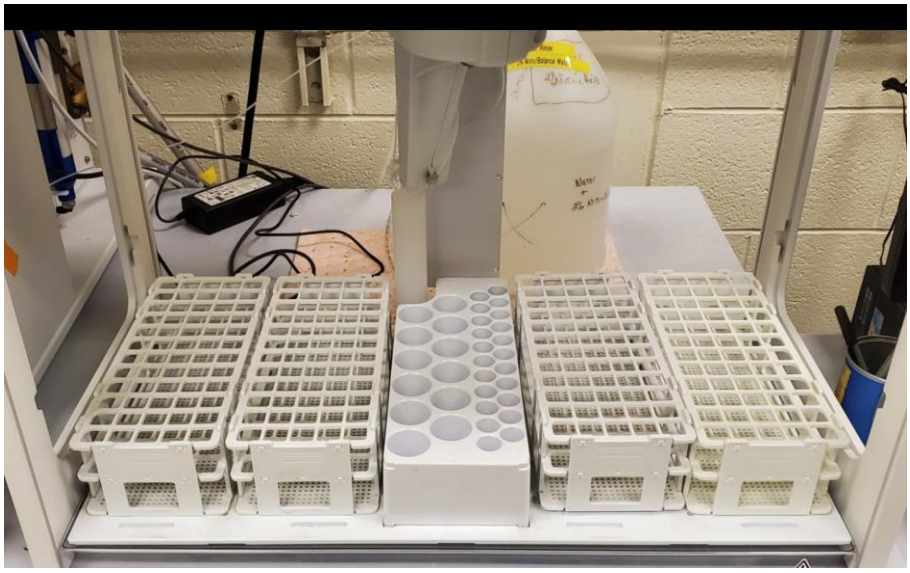

Autosampler

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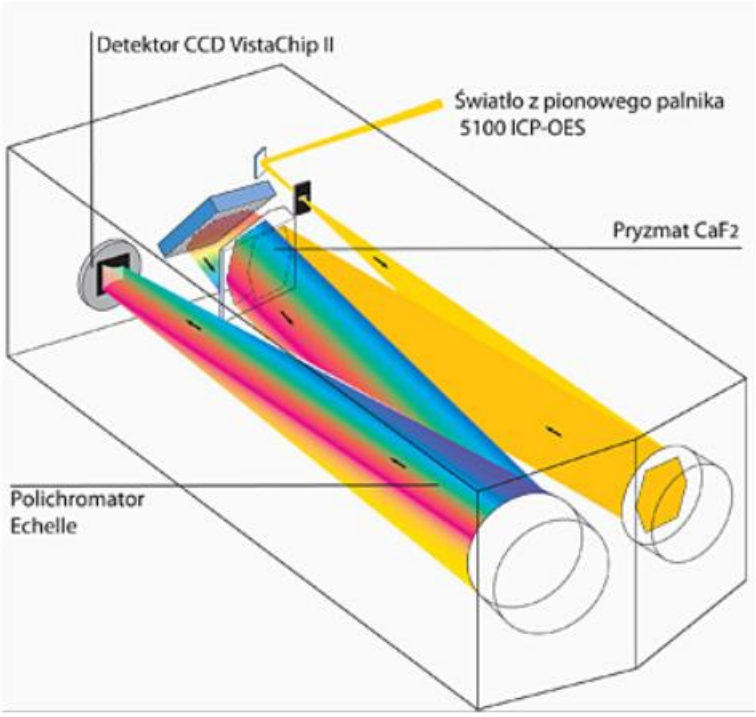

Spectrometer

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Common Conditions

Replicates:	3	
Pump speed (rpm):	12	
Uptake delay (s):	25	☑ Fast pump
Rinse time (s):	30	☑ Fast pump

Measurement Conditions

Read time (s):	5	Nebulizer flow (L/min):	0.70
RF power (kW):	1.20	Plasma flow (L/min):	12.0
Stabilization time (s):	15	Aux flow (L/min):	1.00
Viewing mode:	Radial	Make up flow (L/min):	0.00
Viewing height (mm):	8		

☐ Use multiple conditions

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Preparing for the ICP-OES

Last Updated 20220125 Tim

ICP-OES: Agilent 5100 ICP-OES with DVD.
https://mtim.mit.edu/icp-oes
The sensitivity to most elements is 1-100ppm.

Preparing ICP Samples and standards.
You need to dissolve the elements in solution.
All Samples and Standards must be filtered with a 0.2um filter.
You are expected to arrive at the lab with your samples in the appropriate containers and ready to go.
All Samples and standards must be labeled (No abbreviations).

You will need to bring:
Standards:

At least 5ml of solution per measurement (Axial or Radial).
3-4 Element standards that cover your sample range.
Your "Zero" standard is the sample matrix.
DI Water and 2-5% Nitric or Hydrochloric acid typical to keep elements in suspension.

Samples: At least 5ml per view (Axial or Radial).
Rinse Solution (Optional): At least 200ml or I supply a 2%Nitric Balance DI Water.

Helpful Links:
Inorganic Ventures Education:
https://www.inorganicventures.com/education

Agilent Applications Specialist: Christopher Conklin, Christopher.conklin@agilent.com

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Sample Information - Submit to mtim@mit.edu for approval prior to bringing samples to the lab. A new form is needed anytime your project changes.

*Name:*_____

*Email:*_____

*Date:*_____

Project Overview?
What is the origin of your samples?
What elements are you looking for?
What is your sample matrix?

Standards and Sample Information:

Did you filter all your samples and standards with a 0.2um filter?
Yes No
Do you use any Hydrofluoric Acid in your sample or standards preparation?
Yes No
Are your samples or standards biologically viable?
Yes No
Is your sample radioactive?
Yes No
Does your sample or standards contain any organic solvent?
Yes No
Does your sample Hydrolyze?
Yes No
How much Sodium is in your standards and samples?
Approximate %:_____
How much Total Dissolved Solids?
Approximate %:_____
What is the PH of your sample _____