Phase III of Lab Re-opening
Maximum Room Occupancy: 3 People – 13-4148.
PPE Minimum Requirements: Mask, Gloves & Safety Glasses.
Practice Social Distancing.
Instrument must be reserved 24 hours ahead of time.

Lab Entry
Reserve the instrument 24 hours ahead of time using CORAL.
Put on a new pair of gloves (provided).
Write your username on the lab occupancy white board.
Note: The lab door to 13-4139 is now locked all the time. Contact mtim@mit.edu if you need access with your MIT ID.
Spray the keyboard and work area with Alcohol (Provided).
Engage CORAL

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.
Standard Operating Procedure

Inductively Coupled Plasma – Optical Emission Spectrometer

Last updated 20210112

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
Inductively Coupled Plasma – Optical Emission Spectrometer

Agilent 5100 VDV ICP-OES

https://mtim.mit.edu/icp-oes
Checkout the system when you arrive.
Exhaust Hood Air Flow – Greater than 150 SCFM.
Liquid Argon Dewar – 95 psi delivery pressure, Regulator Valve - Closed.
Waste container – Three drain lines. Good condition, not full, red tag (Undated until full).
Chiller – Off.
Tubing – Loose.
Computer – On.
Software – Closed.
Autosampler – Green Light.
ICP – Yellow Flashing Light.

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”.
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.
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.
Important: Set the Autosampler Probe height to 145

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.
Inductively Coupled Plasma – Optical Emission Spectrometer

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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.

Finishing Up
Disengage CORAL
Spray the keyboard and work area with 70% Alcohol (Provided).
Erase your name from the lab occupancy white board.
Throw your gloves away.
Report any problems, comments or suggestions to: mtim@mit.edu
Inductively Coupled Plasma – Optical Emission Spectrometer

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

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.
**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

<table>
<thead>
<tr>
<th>Wavelength Calibration Solutions</th>
<th>Part Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>ICP-OES wavelength calibration concentrate, 500 mL</td>
<td>6610030000</td>
</tr>
<tr>
<td>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% HNO₃ (Dilute 10x before use)</td>
<td></td>
</tr>
<tr>
<td>ICP-OES wavelength calibration solution, 500 mL</td>
<td>6610030100</td>
</tr>
<tr>
<td>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% HNO₃ (Ready to use)</td>
<td></td>
</tr>
<tr>
<td>ICP-OES calibration blank solution, 500 mL</td>
<td>5190-7001</td>
</tr>
<tr>
<td>Pure ASTM Type 1A water with 5% HNO₃ (For use as a calibration blank or diluent for the wavelength calibration concentrate)</td>
<td></td>
</tr>
</tbody>
</table>
Vendor Contacts
Inorganic Ventures: Standards and sample prep recipes: Amber Compton, a.compton@inorganicventures.com (540) 585-3030

Agilent Applications Specialist: Christine Rivera, Christine.rivera@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, Dr. Mohanraja Kumar, https://cehs.mit.edu/core facilities/bioanalytical-facilities-core/major-equipment
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
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
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.
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.
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.
Inductively Coupled Plasma – Optical Emission Spectrometer

Last updated 20210112

Dual View
Inductively Coupled Plasma – Optical Emission Spectrometer

Last updated 20210112

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
Nebulizer & Spray Chamber
Inductively Coupled Plasma – Optical Emission Spectrometer

Last updated 20210112

Glass Expansion SeaSpray

Nebulizer
Sample Introduction Area
Inductively Coupled Plasma – Optical Emission Spectrometer

Last updated 20210112

Autosampler
Inductively Coupled Plasma – Optical Emission Spectrometer

Last updated 20210112

Spectrometer
# Inductively Coupled Plasma – Optical Emission Spectrometer

*Last updated 20210112*

## Common Conditions

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Replicates</td>
<td>3</td>
</tr>
<tr>
<td>Pump speed (rpm)</td>
<td>12</td>
</tr>
<tr>
<td>Uptake delay (s)</td>
<td>25</td>
</tr>
<tr>
<td>Rinse time (s)</td>
<td>30</td>
</tr>
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</table>

- ✔️ Fast pump

## Measurement Conditions

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Read time (s)</td>
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</tr>
<tr>
<td>RF power (kW)</td>
<td>1.20</td>
</tr>
<tr>
<td>Stabilization time (s)</td>
<td>15</td>
</tr>
<tr>
<td>Viewing mode</td>
<td>Radial</td>
</tr>
<tr>
<td>Viewing height (mm)</td>
<td>8</td>
</tr>
<tr>
<td>Nebulizer flow (L/min)</td>
<td>0.70</td>
</tr>
<tr>
<td>Plasma flow (L/min)</td>
<td>12.0</td>
</tr>
<tr>
<td>Aux flow (L/min)</td>
<td>1.00</td>
</tr>
<tr>
<td>Make up flow (L/min)</td>
<td>0.00</td>
</tr>
</tbody>
</table>

- Use multiple conditions

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Massachusetts Institute of Technology
Materials Research Laboratory
Materials Research Shared Engineering Center
Analysis Shared Experimental Facility
Optical, Thermal Characterization Laboratory
Preparing for the ICP-OES
Last Updated 20200115 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: Christine Rivera:
Christine.rivera@agilent.com
Preparing for the ICP-OES – Worksheet - Submit to mtim@mit.edu for approval

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 ________________