Standard Operating Procedure

Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) - Thermo Scientific iCAP 6300

The Thermo Scientific iCAP 6300 Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) enables trace metal element analysis for liquid samples. It can be used to detect the presence of multi-elements (including non-metals) in a wide variety of samples ranging from food, environment, metallurgy and petrochemical samples with detection limits ranging from sub ppb to % levels.

1.0 Operation Procedure

The ICP-OES is designed to be used by properly trained personnel only. Any adjustment and repair of this equipment must only be carried out by a Thermo Scientific certified field service engineer who is aware of the hazards involved. For the correct and safe use of the instrument and its accessories, it is essential that the operating and service personnel follow generally accepted safety procedures in addition to the specific precautions specified in this SOP.

- Specific warning and caution statements and symbols are marked on the apparatus where appropriate.
- The covers of the instrument and accessories should only be removed by qualified, appropriately trained service personnel.
- All spare parts and consumables items must be approved by Thermo Scientific.
- Some of the chemicals used in the ICP-OES are corrosive and/or flammable, and samples may be radioactive, toxic or potentially infective. Normal laboratory procedures and regulations for handling such materials should be followed.
- All mains powered equipment is designed for operation with a fully earthed mains supply. The mains earth connection to the equipment must be connected, otherwise safety may be impaired.
- If liquid is spilled on or adjacent to the instrument, immediately isolate the instrument and accessories from the electrical supply, by turning off the power remote to the instrumentation.
- Check the ICP-OES manual for appropriate cleaning and decontamination procedures of the equipment if hazardous material is spilt on or inside the equipment.

1.1 Create a method

a. Open the valves of the Ar gas cylinder (max pressure: 6 bar or 90 psi)
b. Turn on the main power switch of the instrument
c. Wait for at least 1 hour to purge the system with Ar gas

d. Turn on the chiller (set point = 18 °C)

e. Click iTEVA software icon on the desktop to launch iTEVA Control Center (user name: admin, no password required)

f. Click Plasma Key in the down menu bar to open Plasma Status dialog box

g. Click “Instrument Status”

h. Wait for: Generator Temperature = 25 °C, Camera Temperature = -46 °C, Optics Temperature = 38 °C. Then, click “Close”.

i. Tighten the tubing attached to the peristaltic pump and pay attention to the direction of rear and front tubing for proper flow

j. Click “Plasma On” to ignite the torch. The system will automatically perform “spectrometer optimization”. After the optimization, the peristaltic pump will start to rotate.

k. Check the parameters in Plasma Status dialog box, then click “Close”:
   - RF Power = 1150 W
   - Pump Rate = 50 rpm
   - Aux. Gas Flow = 0.5 L/min
   - Neb. Gas Flow = On
   - Purge Gas Flow = Normal
1.2 Create a New Method

a. Insert the inlet tubing in DI water, and flush the system with DI water for 10 minutes
b. Insert the inlet tubing in the Blank solution, and flush the system with the Blank solution for 10 minutes
c. Click “Analyst”
d. Select an existing method or create a new method. To create a new method, click Method → New. This will open a periodic table.

e. Choose the elements in grey only and select appropriate wavelength with high relative intensity compared to other elements at the same wavelength. Click OK.

f. Click “Method” tab at the bottom of left panel. It shows a list of settings as below.

g. Select “Analysis Preferences”: # Repeats = 3, Sample flush time = 30 sec, Low WL Range = 15 sec, High WL Range = 5 sec, Mode = concentration

h. Select “Source Settings”: Flush Pump Rate = 100 rpm, Analysis Pump Rate = 50 rpm, Pump Stabilization Time = 5 sec, RF Power = 1150 W for inorganic samples or 1350 W for organic samples, Auxiliary Gas Flow = 0.5 L/min, Radial Viewing Height = 12.0 mm
i. Select “Standards”:

- Keep “Blank” and delete “HighStd”
- Add extra 5 standards (ppm1, ppm2, ppm3, ppm4, and ppm5)
- Enter the value of Default Concentration for each of them (the unit is in ppm)

**NOTE:** the range of these six standard concentration values should bracket the estimate concentration value of your unknown sample

j. To save this new method, click Method → Save As, give the Method Name, and click OK
1.3 Calibrate with Standard

a. Click “Analysis” tab at the bottom of left panel
b. Click “Calibration: Run Calibration Standards” button on the top ribbon

c. Insert the inlet tubing in the Blank solution
d. Select “Blank” in the Calibration dialog box, then click “Run”

e. Insert the inlet tubing in the standard solution named as “ppm1”
f. Select “ppm1” in the Calibration dialog box, then click “Run”
g. Repeat Step 26-27 until all the standard solutions are tested, then click “Done”
h. Click “Method” tab at the bottom of left panel
i. Select “Elements”: it shows the element(s) you selected from the periodic table
j. Select “Fit” tab on the right panel to check the calibration curve of each element
1.4 Test Unknown Samples
   a. Click “Analysis” tab at the bottom of left panel
   b. Click “Run Unknown” button on the top ribbon
   c. Insert the inlet tubing in the unknown sample solution
   d. Input Sample Name in the Run Unknown dialog box, then click “Run”
e. Repeat Step 33-35 until all the unknown samples are tested
f. To export all the unknown sample results, right-click the method name in the left panel, then select “Export All Samples…”

g. Enter the name of the file to be exported
h. Find the exported file in the directory of C:\Users\Public\iTEVA\Export

1.5 Shut Down the System

a. Insert the inlet tubing in DI water, and flush the system with DI water for 10 minutes
b. Click Plasma Key in the down menu bar to open Plasma Status dialog box
c. Click “Plasma Off” to turn off the torch, then click “Close”
d. Loosen the tubing attached to the peristaltic pump and release the tension to increase the tubing life span

e. Leave the chiller on for extra 10 min to cool down the plasma source

f. Close the software iTEVA Control Center

g. Turn off the chiller

h. Turn off the main power switch of the instrument

i. Close the valves of the Ar gas cylinder

2.0 Specifications/Features

- Trace metal element analysis for liquid samples
- Multi-elements detection (including non-metals)
- UHP Ar gas for RF plasma generation
- CID detector with thermoelectric cooling
- iTEVA control software
- Plasma view optics: Radial
- Wavelength range: 166 to 847 nm
- Detection capability: sub ppb to % levels (for most elements when measured at their optimum wavelength)

3.0 User Requirements

The Thermo Scientific iCAP 6300 ICP-OES must be used by authorized personnel only. All authorized users are expected to read and understand this SOP and follow the operation instructions carefully. No unauthorized user may operate this ICP-OES unless accompanied by an authorized user. All visitors must be briefed on proper safety protocol and must wear appropriate personal protective equipment. To become an authorized user, one must:

1. Complete Environment, Health & Safety (EH&S) training

2. Complete initial orientation and training for the Materials Characterization Laboratory

3. Receive training on this piece of equipment from lab personnel

4. Schedule equipment time using the calendar

5. Read and fully understand this SOP
4.0 General Safety

4.1 Required Personal Protective Equipment
Users must wear lab coats, safety glasses, and gloves. Shorts, open-toed shoes, high heels, and skirts, are forbidden.

4.2 Emergency Procedures and Contacts
For non-life threatening emergencies: notify the MCL facility manager and your PI immediately.
Facility manager: Zongmin (Shirley) Bei, Ph.D.
Office: 109B Furnas Hall, Tel: (716) 645-5165, Cell: (585) 354-5623
Email: zongminb@buffalo.edu
or for police / ambulance, call 645-2222

In case of fire or other life threatening emergency: Exit the laboratory through an emergency exit door. Pull one of the fire alarms located in the main hallway. Dial campus police / ambulance at 645-2222.

4.3 University after hours laboratory use policy
No working alone, use the buddy system!

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