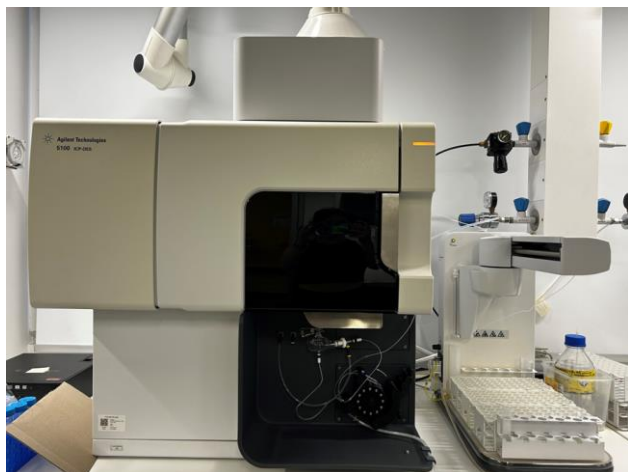


STANDARD OPERATING PROCEDURE

[Inductively Coupled Plasma Optical Emission
Spectrophotometer]

Doc No. ICPOES-v1.1

Date: Dec 2024
Review Date: Dec 2026
Version No. 1.1
Authorised by: Alex Duan



Instrument: ICP-OES

Brand/Model: Agilent Technology/5100 ICPOES SPS3

Software: ICPEXpert

1. INTRODUCTION

Inductively Coupled Plasma Optical Emission Spectrophotometer (**ICPOES**) is a solution-based technique whereby samples are prepared and introduced in aqueous (liquid) forms for analysis.

With utilization of an argon plasma (as high as 8000°C), the atoms and/or ions are excited to emit light (photons). By measuring the intensity of the emitted light, which is characteristic to each element, the concentration of particular element(s) can be derived from a calibration curve.

2. SCOPE

The scope of this standard operating procedure (SOP) outlines the safe use and operation of Agilent 5100 ICPOES with a SPS3 autosampler. The SOP will describe operation and analysis utilizing the ICPEXpert software to control the ICP-OES and autosampler. This is considered a low risk item.

3. REQUIREMENTS

3.1 Roles and responsibilities

Instructor is a TrACEES Platform staff who has the adequate expertise and training responsibility for the instrument.

Instrument supervisor is the Platform staff who is responsible for maintenance and troubleshoot.

Operators (or Users) are any internal (incl. the University's students and staff) and external personnel's.

3.2 Personal Protective Equipment (PPE)

Laboratory coat, safety glasses, fully covered footwear, gloves when handling toxic materials.

3.3 Training and Induction

Prerequisites TrainMe Modules:

PPE, Laboratory Safety and Chemical Management.

All users shall complete a local lab induction and be trained by the instructor.

Users from Schools/Department other than School of Chemistry shall also complete a building induction.

4. WARNINGS

Extreme temperatures (plasma can reach up to 8000°C).

Production of noxious fumes.

Handling corrosive acids or bases.

Sample solutions must be filtered through a 0.45µm syringe filter.

5. OPERATION

5.1 Start Up

5.1.1 Start Up

Turn on extraction fan.

Turn on water chiller.

Ensure nitrogen and argon gases are turned on

Ensure Autosampler is on. On button is at the back right side of the autosampler.

5.1.2 Tubing and Pump setup

Gently stretch and hook the tubing around the pump rotor using the hook points ensuring the tubing lines up with the groove.

Lift the clamp of the corresponding section and clamp the tubing in place.

Repeat hooking and clamping with pump tubing located

on the left side of the autosampler.

Check wash solution is full. If not using the washing solution currently connected, remove the tubing from the bottle of washing solution, rinse the tubing over a beaker with MillQ water (MQW) and transfer into new washing solution.

5.1.3 Open the software (ICPEXpert)

On the computer's desktop, locate '**ICPEXpert**' icon and double click.

5.2 During Operation

5.2.1 Instrument warm-up procedure

Open '**Instrument Panel**'. Ensure all systems have a green light on the status panel located on the left-hand side.

Turn '**Plasma ON**' from the toolbar at the top of the screen. Check plasma has ignited.

Open '**Autosampler**' menu and move the autosampler to MQW by double clicking on the location containing a MQW blank.

NOTE Plasma needs to stabilize for 20 minutes prior to analysis.

5.2.2 Creating a new method

Create a new worksheet by double clicking on '**New**' on the main screen.

Navigate to the '**Elements**' tab and choose elements for analysis by double clicking on the element in the periodic table or selecting from the drop-down menu. Choose an appropriate wavelength for analysis and click add. It is suggested for new samples to select the top three wavelengths for analysis.

Select '**Enable QC**' under the periodic table.

Navigate to the '**Conditions**' tab. Select number of replicates and viewing mode. If multiple conditions are required tick '**Use multiple conditions**' and create two condition methods. When multiple methods are required, they will need to be allocated to the samples on the left-hand side.

Navigate to the '**Standards**' tab and select number of standards. Add the standards name and concentrations of analytes of interest.

In the '**QC**' tab add '**CCB**' and '**CCV**'.

Navigate to the '**sequence**' tab and enter samples for analysis ensuring to change the sample positions.

NOTE It is necessary to add a sequence of CCB and CCV every 15 samples to monitor for drift. This can be added by right clicking in the sequence selecting add solution below and selecting the required QC standard.

Save the worksheet with a unique data file in appropriate folder. Suggested format "[YYMMDD]-[Operator Initial]-[Run]", e.g. "231120-AD-run1"
Do Not use "space" or any special characters.

5.2.3 Analysis of samples

Load samples into the autosampler trays ensuring there is at least 2 mL for one condition analysis and 3-4 mL for two condition analysis.

In the '**Autosampler**' tab confirm samples have been entered and loaded into the correct locations.

In the '**Autosampler**' menu move the autosampler to '**Park**' and '**Rinse**' until the autosampler begins draining to the waste and return to '**Park**'

Move to the analysis window ensure all samples which require analysis are ticked and click '**RUN**' in the top toolbar.

NOTE Prior to injection samples must be filtered with a 0.45 µm syringe filter.

5.3 Shut Down

Wash autosampler with washing solution for 2-3 minutes by moving the autosampler to '**Rinse**' via to '**Autosampler**' menu. (**NOTE**: Keep plasma on for 3 minutes while in washing position)

Move autosampler to '**Park**' in '**Autosampler**' menu.

Unlock arms and release tension in tubing on peristaltic and autosampler pump.

Turn off plasma.

A few minutes after the plasma has been extinguished the water chiller may be turned off.

Close down ICPEXpert program.

DO NOT turn off ICP, nitrogen or argon gases or exhaust fan.

5.4 Clean up

Users should remove all samples from the autosampler at the completion of their session and dispose of at their own labs.

5.5 Data transfer

Users shall not bring and use own data storage device such as USB sticks.

Mediaflux Data Mover is the preferred method.

6. MAINTENANCE/TROUBLE SHOOTING

Operators shall keep the working area clean and tidy.

Operators are not required for instrument maintenance and trouble shooting.

Contact the instrument supervisor for any equipment related issues.

7. INCIDENT/EMERGENCY

7.1 Incident

In the event of a minor incident (spill and cuts), operators shall report to the instrument supervisor or lab manager immediately.

Chemical spills: users shall use spill kit and follow clean up procedure.

Any contact with chemicals, wash the area immediately, and use emergency showers or eyewash for more serious incidents.

Minor cuts: use first aid kits properly if needed.

First aid assistance shall be provided by the area's first aid officer.

7.2 Emergency

In the event of a major incident/emergency (fire, explosion, medical emergency), operators shall:

- Notify the relevant emergency response department e.g. Fire, Ambulance, Police (dial "000").
- Notify the School of Chemistry OHS Coordinator (03 90355494)

8. REFERENCES

8.1 Legislation

Occupational Health and Safety Act 2004

Occupational Health and Safety Regulations 2007

8.2 Codes and Standards

AS/NZS 1337.1-2: Personal eye-protection

AS/NZS 1337.4-6: Eye and face protection

AS/NZS 2161 (Series): Occupational protective gloves

8.3 University Procedures/Guidance

The equipment is not considered as Regulated Plant.

See OHS' Regulated Plant Requirements

(<https://safety.unimelb.edu.au>)

