
	<p style="text-align: center;"><i>Standard Operating Procedure</i></p> 	Effective Date:	Version:
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Standard Operation Procedure – Microwave Plasma Atomic Emission Spectrometer

1. Scope and Application

Operation of the Agilent 4100 MP-AES involves the use of compressed air, high microwave energy and hazardous materials including corrosive fluids and flammable liquids. The plasma is extremely hot (about 6000°C) and operates using high levels of microwave energy. The plasma emits high intensity light. The microwave excitation assembly is designed to reduce microwave radiation to safe levels while still allowing easy installation of the torch and viewing of the plasma.

The various indicator lights are colour coded to represent the status of the instrument.

- **A green light indicates the instrument is in normal/standby mode.**
 - **An orange light indicates that a potential hazard is present.**
 - **A blue light indicates that operator intervention is required.**
 - **A red light warns of danger or an emergency.**
 - The primary gas used is nitrogen, which is the supply gas for the plasma and nebulizer gas supply Instrument grade quality required.
 - A small quantity of argon is used only in the plasma ignition cycle.
 - Oil free compressed air is used for the pre optics protection gas.
- Pic page 24 and 25.

2. Sample preparation

- Weigh a well-mixed sample to the nearest 0.001g in the digestion vessels. For sludge, faecal samples use between 0.1g and 0.5g. For oil or oil contaminated samples use no more than 0.25g.
- If the sample cannot be well mixed and homogenised then oven dry at 60°C or less and then grind the sample.
- Add 12ml of Aquaregia (9ml conc nitric acid + 3ml conc hydrochloric acid) to the samples in the digestion vessels.
- Dilute each sample to 50ml (then centrifuge) and test using the MP-AES.
- The microwave program is set as follows:
 - 04:00min @1000w@90°C
 - 05:00min@1000 w @130°C

- 04:00min@1000 w @190°C
- 10:00min@1000 w @190°C
- 30:00min@1000 w @30°C

Total time 1:03:00

- 04:00min @1000w@90°C
- 60:00min@1000 w @130°C
- 10:00min@1000 w @40°C
- 30:00min@1000 w @30°C

3. To install the MP Expert Software

1. Log on to the instrument computer with Administrative rights. (username-Admin/password-3000hanover).
2. Insert the MP Expert Software disk in your CD Rom drive. The software will automatically start.
3. Follow the instructions on the screen.
4. Click Yes to restart the computer if prompted.
5. Plug the USB cable into the USB port and then into a USB port on the computer.
6. Once installation is complete, you will need to register your software. To start the software, click the Windows START button, Then ALL PROGRAMS>Agilent>MP Expert.
7. Windows START button then choose Programs>Agilent>MP Expert>MP Expert Help.

Analysis Checklist

- Turn on the MP-AES and software
- Prepare for analysis
- Calibrate the MP-AES
- Create/open a worksheet
- Develop a method
- Run samples
- Print a report

Turning on the MP-AES and Software

1. Check the exhaust and intake lines are secured to the MP-AES.
2. Ensure the gas lines are connected to the MP-AES and the gas supplies are turned on and set to the correct pressures (4-6 bars). Switch on the nitrogen gas generator.
3. Check that the USB and power cables are plugged on.
4. Switch on the compressor and the extractor system.

Instrument

5. Turn on the MP-AES.
6. Switch on the computer(username-Admin/password-3000hanover)
7. To start the MP Expert software, click the Windows start button and then choose Programs>Agilent>MP Expert>MP Expert. The main index window will appear.

8. Click on instrument.
9. *Red blinking zones on the instrument model on the pc stipulates errors. Check the error bar reading on the left. Purge the instrument to remove O₂.*
10. Click-Start Purge(1-2mins to purge)
11. Blinking should stop after purging.

Torch

12. Check that the pre optics window is clean and correctly installed and that the interlocked is engaged.
13. Insert the torch with the outstanding lever facing you and completely close the torch handle. Do not touch the torch-this will create hotspots. Fit the spray chamber socket to the ball joint on the base of the torch and secure using the torch clamp. Monitor pic on pc-red zones will appear if it is inserted incorrectly.

Pump

14. Check that all tubing on the spray chamber, nebulizer and peristaltic pump are correctly connected. See pic.
15. Inlet tubing should be placed in a beaker of 500ml de-ionised water.
16. Go to software-Click on –Run pump icon (the lower icon).
17. Click on fast run (run for 2 mins) and look for a flow.
18. Click on Pump Off.
19. Check icons on left are all green.

Plasma

20. Ensure that the Plasma Enable Switch is in the Enable state (pushed in).
21. Click-Plasma On(6000 degrees Celsius)
22. Inspect flame-must taper uniformly. Must have only the following colours (orange on the outside-pink and the blue in the middle).
23. Switch off if it is not correct.
24. Leave the plasma on while continuing with the calibration.

Preparing for Analysis

1. Click the Plasma button in the MP Expert software, press F5 or choose Plasma on from the arrow under the plasma button.
2. Ensure that the peristaltic pump is correctly set up.
3. Place the sample tubing from the peristaltic pump into the rinse solution and the drain tubing into the drainage vessel.
4. Click the pump button in the MP-Expert software and choose Normal (15rpm) from the arrow under the pump button. The pump will be initialized and the solution will begin aspirating.
5. It will take approx. 30 mins for the MP-AES to warm up.
6. The plasma will take 10-15 secs to ignite. If it fails, refer to the troubleshooting guide.

7. The plasma cannot run without the spray chamber and the nebulizer gas supply connected. Doing so will damage the torch.

MP-AES Calibration

1. Ensure a standard glass concentric nebuliser; a single pass spray chamber and a standard plasma torch are installed. Use white/white peristaltic pump solution tubing and blue/blue tubing for the drain. The tubing tab colour denotes the tubing size.
2. Place the solution inlet tubing into the wavelength calibration solution and allow the sample to reach the plasma.
3. Click the instrument button.
4. Click Optics Calibration.
5. Click Calibrate Instrument. The torch will be aligned then a wavelength calibration and a calibration check will be performed automatically.
6. After a short while, an indication of the success or failure of the calibration check will appear, as well as an indication of the wavelength offsets.
7. If the calibration fails, check the sample introduction system. If the system seems fine, prepare a new wavelength calibration solution and try again.
8. Recommended values for the settings are given on the Conditions.

Test

Tick all tests except the last.

Click RUN TEST

Export to pdf and save.

Creating/Opening a Worksheet

Click NEW from the START page or the FILE menu.

A list of recently used files will appear otherwise you may BROWSE for more.

Opening an existing worksheet.

- Click OPEN from the Start page or the File menu.
- A list of recently used files will appear otherwise you may BROWSE for more. The OPEN dialog box will be displayed in this instant.
- **Choosing rack type**
Autosampler right click/Rack type/no rack
Go to bottom rack/right click/rack type/e.g. 11 samples.
Creating a new worksheet from a template
- Click NEW Form on the Start page or New Form Template from the file menu.
- A list of recently used files will be presented; otherwise, you may BROWSE for more files. The New Form Template dialog box will be displayed in this instance.
- The Worksheet window will appear with the new worksheet loaded.

Running a sample

1. Click the ANALYSIS tab and do the following.
2. Ensure your samples are selected. This will be indicated by a check next to the RACK: Tube column. To select all solutions, select the checkbox next to the Rack: Tube title.
3. Click the RUN icon in the toolbar (or PRESS the SHIFT+F8) to begin the analysis, and follow the subsequent prompts.

Printing a report

Click Report on the toolbar or file>Report

Choose whether you want to print or preview the report or save the report as a PDF file.

Select the report template and click OPEN.

Click the PRINT button to generate a report as specified.

Turning off the Agilent 4100 MP-AES

- Rinse the spray chamber by aspirating water for a few minutes. 1% nitric acid.
- Extinguish the plasma by choosing 'PLASMA OFF' from the Plasma button drop down arrow or pressing SHIFT +F5 on the keyboard.
- Remove the solution tubing from the solution to prevent flooding of the spray chamber. Choose (normal-15 rpm) from the pump drop down button and pump any remaining liquid from the sample introduction system and inlet tubing. Turn the pump off when there is no more liquid flowing.
The green Plasma enable button, located in front of the MP-AES is intended for emergency only. If it is used you will have to reset the plasma enable button to 'on' position before the plasma can be re-ignited.
- **To increase the pump tubing lifetime, loosen the pressure on the peristaltic pump tubing by releasing the pressure bars, and lifting the tubing out of the grooves in the tube retainer. PIC page 39.**
- **Push up the pressure bar tensioners. This releases them from the pressure bars.**
- **Allow the pressure bar to swing backwards.**
- **Lift the tubing out of the grooves in the tube retainer.**

APPROVAL OF STANDARD OPERATING PROCEDURE

PRG Head: Prof C.A. Buckley

Signature:

Date:

Author: Merlien Reddy

Signature:

Date: