



Standard Operating Procedure

Gas Chromatograph Mass Spectrometry (GC/MS) - Agilent 7890B GC & 240 Ion Trap MS

The Agilent 7890B is a state-of-art gas chromatograph (GC) that provides superior performance for all GC applications with the use of advanced electronic pneumatic control (EPC) modules and high performance GC oven temperature control. The Agilent 240 Ion Trap mass spectrometer (MS), combined with the Agilent 7890B GC, delivers exceptional performance and unsurpassed flexibility with both MS and GC options. The MS is configured in internal mode and provides both electron ionization (EI) and chemical ionization (CI). The 7693 automatic liquid sampler (ALS) works as the interface on 7890B, providing power and communications for one automatic injector and one automatic sampler tray that are easily installed without the need for alignment.

1.0 Operation Procedure

Power outage: If there is a power outage, turn off the GC-MS according to the shutdown procedure to avoid a hazardous situation when power is restored. If there is an emergency, leave the MCL immediately and either return after emergency to shut down the GC-MS or contact the facility manager.

When handling/using chemicals for preparation or use within the GC & MS, all applicable local and national laboratory safety practices must be followed. This would include, but is not limited to, correct use of Personal Protective Equipment (PPE), correct use of storage vials, and correct handling of chemicals. There are several important safety notices that you should always keep in mind when using the Agilent 7890 B GC & 240 Ion Trap MS:

- **Many internal parts of the GC & MS carry dangerous voltages**

If the GC & MS are connected to a power source, even if the power switch is off, potentially dangerous voltages exist on:

- ✓ The wiring between the GC & MS power cords and the AC power supply, the AC power supply itself, and the wiring from the AC power supply to the power switch.

With the power switch on, potentially dangerous voltages also exist on:

- ✓ All electronics boards in the instrument.

- ✓ The internal wires and cables connected to these boards.

- ✓ The wires for any heater (oven, detector, inlet, or valve box).



All these parts are shielded by covers. With the covers in place, it should be difficult to accidentally make contact with dangerous voltages. Unless specifically instructed to, never remove a cover unless the detector, inlet, or oven are turned off.

- **Do not use an Uninterruptable Power Supply (UPS) with a GC**

If the area where the GC is located suddenly loses power, an unsafe condition can result if the GC remains powered on. Do not use the GC with a UPS.

- **Electrostatic discharge is a threat to the GC & MS electronics**

The printed circuit (PC) boards in the GC & MS can be damaged by electrostatic discharge. Do not touch any of the boards unless it is absolutely necessary. If you must handle them, wear a grounded wrist strap and take other antistatic precautions. Wear a grounded wrist strap any time you must remove the GC right side cover.

- **Many parts are dangerously hot**

Many parts of the GC & MS operate at temperatures high enough to cause serious burns. These parts include but are not limited to: the inlets, the GC oven and its contents, the MS ion trap oven, the MS manifold, the MS transfer line, the GC heated interface, the column nuts attaching the column to an inlet or detector, the valve box, and the MS foreline pump.

You should always cool these areas of the GC & MS systems to room temperature before working on them. They will cool faster if you first set the temperature of the heated zone to room temperature. Turn the zone off after it has reached the setpoint. Be careful when working behind the instrument. During cool-down cycles, the GC emits hot exhaust which can cause burns.

- **Oven thermal leaks**

Objects passing through the oven door seal can cause thermal leaks which create hazardous hot spots which cause burns and melt equipment. Do not allow wiring or temperature probes to pass through the oven door jam. Agilent recommends using one of the access holes.

- **Micro-Electron Capture Detector (μ ECD) on GC**

The μ ECD on GC system contains a cell plated with ^{63}Ni , a radioactive isotope. The beta particles released at the energy level in the detector have little penetrating power – the surface layer of the skin or a few sheets of paper will stop most of them – but they may



be hazardous if the isotope is ingested or inhaled. For this reason, handle the cell with care. Cap the detector inlet and outlet fittings when the detector is not in use. Never introduce corrosive chemicals into the detector. Do not use solvents to clean the μ ECD.

Materials that may react with the ^{63}Ni source, either to form volatile products or to cause physical degradation of the plated film, must be avoided. These materials include oxidizing compounds, acids, wet halogens, wet nitric acid, ammonium hydroxide, hydrogen sulfide, PCPs, and carbon monoxide. This list is not exhaustive but indicates the kinds of compounds that may cause damage to ^{63}Ni detectors.

When handling the μ ECD on GC system:

- ✓ Never eat, drink, or smoke.
- ✓ Always wear safety glasses when working with or near the open μ ECD.
- ✓ Wear protective clothing such as laboratory jackets, safety glasses, and gloves, and follow good laboratory practices. Wash hands thoroughly with a mild nonabrasive cleaner after handling the μ ECD.
- ✓ Cap the inlet and outlet fittings when the μ ECD is not in use.
- **The oil pan under the standard MS foreline pump can be a fire hazard**
Oily rags, paper towels, and similar absorbents in the oil pan could ignite and damage the pump and other parts of the MS. Combustible materials (or flammable/non-flammable wicking material) placed under, over, or around the foreline (roughing) pump constitutes a fire hazard. Keep the pan clean, but do not leave absorbent material such as paper towels in it.

1.1 Refill Wash Vial

- a. Check and refill wash vials on the injector tower (Fig. 1). The volume of solvent should be above the minimum level specified on the wash vial.
- b. Default wash vials: A: dichloromethane (DCM); B: methyl *tert*-butyl ether (MTBE). Your method specifies which wash vials will be used. For all ECD methods, use wash vial B. Refill solvent based on your method.

NOTE: See separate document for GC-MS method development.

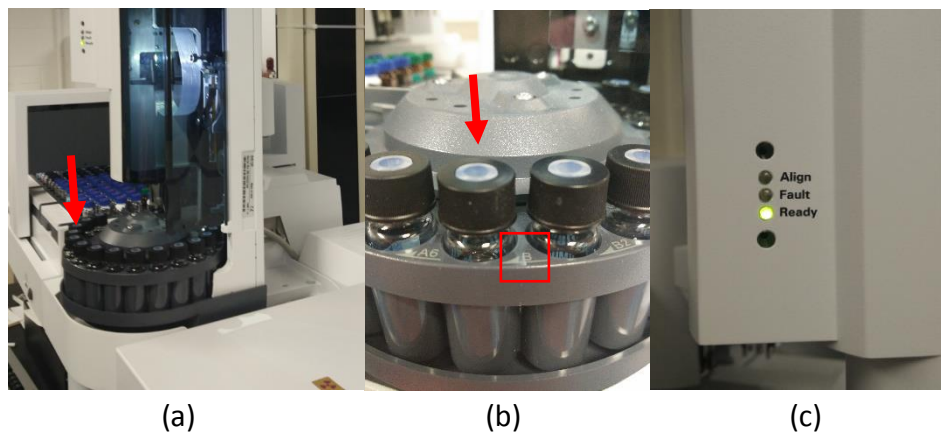


Figure 1 (a) Injector tower; (b) Wash vials; (c) Indicator light

1.2 Switch Injector Tower

- Mount the injector tower over the injection port specified in your method. Default injection ports: Front: mass spectrometry (MS); Back: electron capture detector (ECD).
- Ensure that the *Ready* light is on after switching the injector tower (Fig. 1-c).
- Restart the GC system. See the Step #9 in Section 5.4.

1.3 Load Samples

- Push the button “P” (parking) to move the auto-sampler arm to the left (Fig. 2-c).
- Put your samples in the auto-sampler tray and record the sample number (Fig 2-b).
- Push the button “P” again. Sample injection will not start when the auto-sampler at the parking mode.

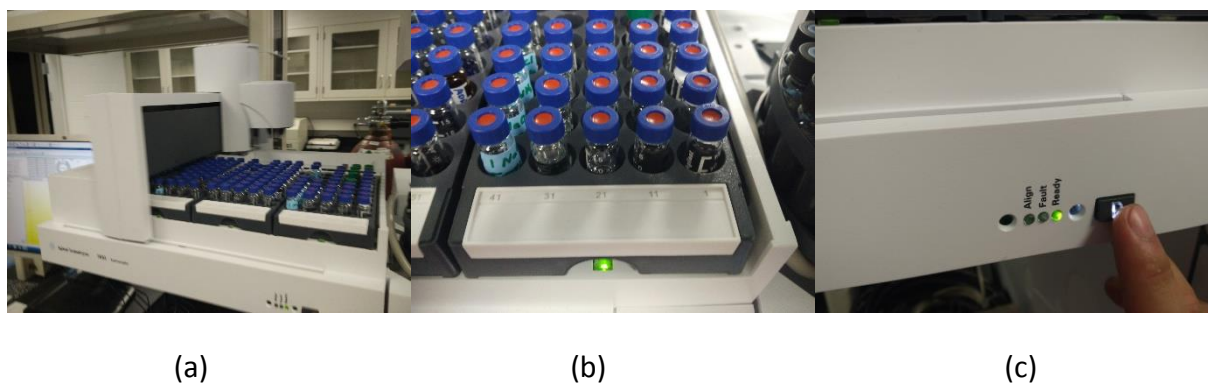


Figure 2 (a) Auto-sampler tray; (b) Sample number; (c) Button “P”

1.4 Software Control

- a. Click “System Control” (Fig. 3) to open control software (Fig. 4).



Figure 3 The button to turn on the control of GC system

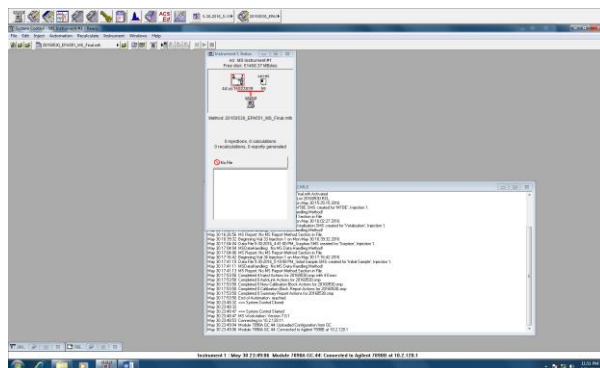


Figure 4 Instrument control software interface

- b. Click the minimized window of “7890A GC.44” (Fig. 5) to open the operation window (Fig. 6).



Figure 5 Minimized window of “7890A GC.44”

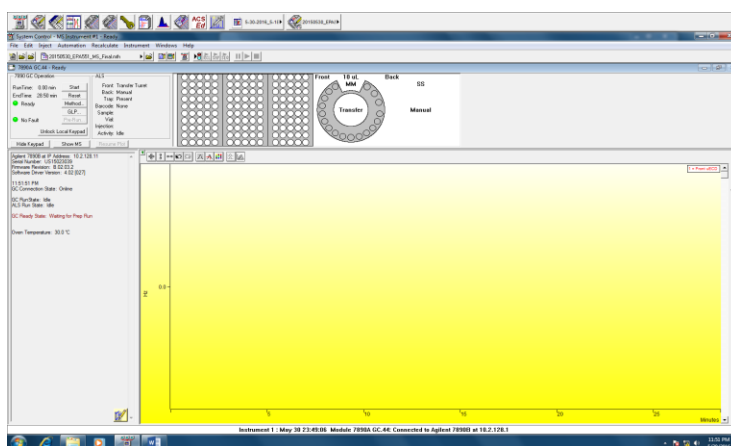


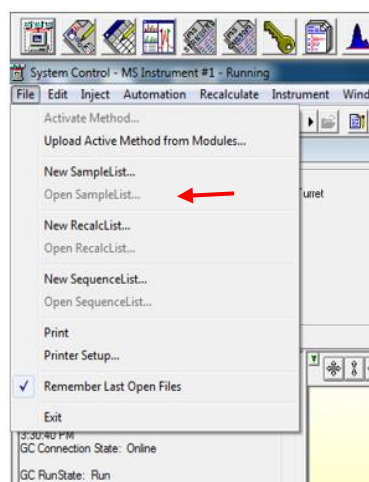
Figure 6 GC operation window

- c. Load your method by clicking the icon at the right side of method name (Fig. 7).

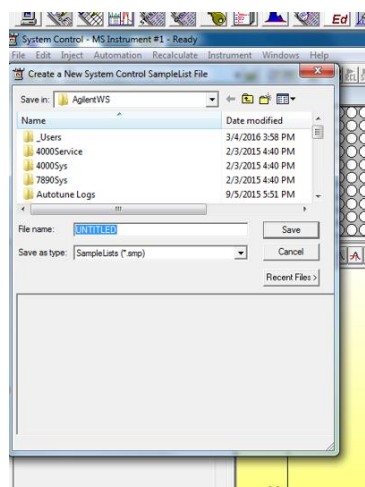


Figure 7 The icon to load your method

- d. Set up sample list
- ✓ Click “File” → “New SampleList” to open the new window (Fig. 8).



(a)



(b)

Figure 8 (a) “New SampleList” and (b) the new window

- ✓ Find the folder with your name. If this is your first time using this GC, add a new folder under the profile of “_Users” with your name as the folder name.
- ✓ Add a new folder named with the date (for example: 20160531) in your folder.
- ✓ Enter the date as the name of your new sample list, and save it under the folder named with the same date.
- ✓ Click “Save” to add the new sample list.
- ✓ Enter sample name with vial number (i.e., sample position on the auto-sampler), injection volume, and injector position (Fig. 9). Note that your injection volume should match the value specified in your method.
- ✓ It is recommended to include a solvent blank as your first sample.

	Sample Name	Sample Type	Cal. level	Inj.	Injection Notes	AutoLink	Vial	Injection Volume	Position	Sample Prep Program	Amount Std (IS, N% only)	Unid Peak Factor	Multip
1	MTBE	Analysis		1	none	none	31	3.00	Front	none	1	0	1
2													
3													
4													

Figure 9 The four items need to fill

- e. Save the data files by clicking “Data File”. The data files should be saved under the folder named with the date. Do not choose this folder as the default directory.
- f. Start running samples by clicking “Begin” and twice “OK”. In addition, the method should be double-checked in the new window.
- g. Ensure to fill the log-in sheet to record the start/end time as well as the parameters

1.5 Post-run Cleanup

- a. Take out your samples from the auto-sampler after detection. Refer to Section 5.3 to move the auto-sampler arm.
- b. Click the cross icon on the top right corner to shut down the control software.
- c. Data analysis for your samples will depend on your method.

NOTE: See separate document for GC-MS method development.

2.0 Specifications/Features

- Three compartments:
 - 7890B GC System (G3440B)
 - 240 Ion Trap MS System (G3934A)
 - 7693 Automatic Liquid Sampler (G4514A)
- 7890B GC System:
 - Retention time repeatability < 0.008% or < 0.0008 min
 - Area repeatability < 1% RSD
 - Pressure setpoint and control precision to 0.001 psi
 - Column oven temperature: ambient +4 °C to 450 °C
Setpoint resolution: 0.1 °C
Maximum ramp rate: 75 °C
Cool down (22 °C ambient) from 450 °C to 50 °C in 4 min
 - Reliable, leak-free, in-oven capillary connections by Capillary Flow Technology
 - Micro- Electron Capture Detector (ECD) with EPC module
*Radioactive material: use < 15 mCi Ni-63 as the electron source



- Multimode inlet (MMI) and Split/Splitless capillary inlet (S/SL) with EPC module
- Carrier gas and auxiliary gas: He
- Detector gas: argon/methane mixture
- 240 Ion Trap MS System:
 - Mass range: 10-1000 Da in 0.1 Da steps (time programmable throughout the run)
 - Scan rate: 5000 or 10000 Da/sec
 - Mass axis stability: ± 0.1 Da over 72 hr
 - Operation temperature: up to 250 °C for mass analyzer, up to 120 °C for manifold
 - Electrode surface: SilChrom for maximum inertness
 - Ionization filament current: 10 – 100 μ A
 - Maximum ionization time: 65 msec
 - Internal mode electron ionization (EI) and chemical ionization (CI)
 - Detection system:
 - Detector: off-axis design, ± 10 kV HED and electron multiplier
 - Analytical linear dynamic range: 10^3 to 10^4
 - Vacuum system:
 - 280 L/s turbomolecular pump
 - Bayard-Alpert ion gauge with thoria-coated iridium filaments
 - DS-102 rotary vane foreline pump, > 95 L/min
 - Foreline pump thermal vacuum gauge
 - Control software: MS Workstation (MSWS)
 - NIST 2014 MS Library Bundle including 243,000 spectra with names, chemical structures, and retention indices

3.0 User Requirements

The Agilent 7890B GC & 240 Ion Trap MS 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 personnel may use this equipment. All users 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!

#	Revised by	Date	Modification
1	Jiale Xu / Ning Dai	06/09/2016	Document initial release
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