# Protocol for quantifying CO2 in headspace samples with the Shimadzu GCMS-QP2010

## Authorship

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# Equipment

* GCMS
  + Shimadzu GCMS-QP2010
* Autosampler
  + Shimadzu AOC-5000 Auto Injector

# GCMS start up

## WARNINGS:

* You should not need to start the floor vacuum pump (rotory pump) manually. This can be done using the GCMS software.
* If the GCMS has not been used in a while:
  + Check the oil level in the rotory vacuum pump (on the floor).
  + Check the amount of carrier gas still in the tank.

## Procedure:

1. Turn on carrier gas (should be Helium)
   * The PSI should be ~100
2. Turn on Shimadzu AOC-5000 Auto Injector
3. Turn on computer
4. Turn on both the GC and the MS
5. Start the **GCMS Real Time Analysis software**

# The next steps are done in GCMS Real Time Analysis software

1. Select **Vacuum control**
2. Use the manual startup ('Vacuum control' => 'advanced...')
   * Manual startup provides more control, option for safety checks, and may produce less wear and tear.
   * Manual startup procedure:
     1. Start the flow controller and GC system
     2. Close vent valve
     3. Turn on rotory pump
        + Let the pressure drop to <3.1 pascals
        + This takes many minutes
     4. AFTER pressure is <3.1 pascals: turn on high pressure pump (ie., the turbo molecular pump)
        + **Note:** A sound resembling a jet engine is normal, as long as it only lasts < a minute.
     5. Turn on ion source heater
        + GC and MS indicators in top right of screen should soon say 'ready'
3. Load a method or create a new method
   * Make sure that GC and MS are heating up to the method's specified temps
   * A batch file can then be created if needed

# GCMS tuning

## WARNINGS:

* If the GCMS has not been used in a long time (e.g., a year), wait ~1 day prior to tuning.
  + This wait provides time for the column to be purged of contaminants.

## Procedure:

* Follow procedure starting on Page 21 of the GCMS-QP2010 Operation Guide.

### Leak check:

* Follow procedure outlined on Page 19 of the GCMS-QP2010 Operation Guide.

# GCMS sample processing

## Notes:

* The septum can be changed while the GC is on. The carrier gas flow will just need to be turned off very briefly prior to changing the septum.

# GCMS shut down

* Use manual shutdown.
  + Auto shutdown does not give enough time between the turbo molecular pump shutdown and the vacuum pump shutdown.
* Wait for the GC to cool before turning off the carrier gas.

# General notes

* The blue septa may provide a better seal than the green septa (for higher temps).
* The rotory vacuum oil should be changed every 6 months.
* Replate the injection port glass liner (contains glass wool) after ~1000 injections.
  + This helps prevent the liner from fusing to the injection port.

# Maintenance

## Autosampler

* Service is need every ~2 years.
* Do not lubricate anything besides the guide rails in the horizontal portion.
  + Lubricating other components will likely break them.
* You can find videos on how to perform maintenance by googling 'leap autosampler'.

# Notes on Carboxen 1010 PLOT column

* Bake at 200oC for 1-2 hours if column disconnected for < 1 day.
* If it is diconnected for >1 day, bake overnight at 200oC.