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# General Operating Procedure for:

# GC-MS (7890B+5977A)



## Department of Health and Environmental Sciences

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# Contents

## 1. Purpose

The following document provides instrument for GCMS routine use.

## 2. Scope

This SOP applies to all personnel using and managing GCMS.

## 3. Sample preparation

A. Sample requirement: GC-MS can only analyse volatile and thermally stable compounds. Basically, solid particles, sugars, proteins, high mass polymers, non-volatile compounds and other similar compounds **MUST NEVER** be injected in the system.
B. Solvent selection: Water is detrimental to the GC column so always make sure that the solvent is very dry. Also the low boiling solvents (i.e. bp <30) may also be problematic.
C. Sample concentration: The concentration of sample must be as low as possible, but higher than the lower limit of detection. Some variation in the concentration can be dealt with by adjusting the split ration of the injector.
D. Filtration: Sample needs to be filtered with 0.2 µm filters or by other sample clean up approaches before injection.

## 4. Getting started

A. Gas cylinder: Before injection, users must make sure that the helium cylinders in ES436B have enough gas supply for the running (*Fig.1*). The outlet pressure setting is 5 bar, no need to change. Put the black valve to horizontal position to start the gas supply for GCMS (*Fig.2*).

**Fig.1**

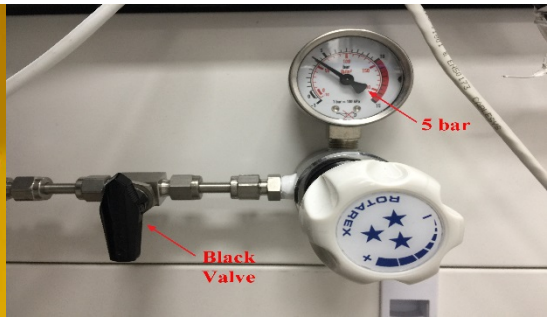



**Fig. 2**

B. Washing solvents and waste vial level: Check the valves in auto sampler tower and ensure that there is an adequate quantity of washing solvent (usually the same as sample preparation solvent) in washing vial. Empty the waste vial if it is over half full.

C. Power on the instrument. Push the panel of the MS to help creating the vacuum (*Fig.3*). Power on the MS module. It will takes about 20mins for the speed of Tubo pump to reach 85% if the ISSon. Power on the GC module. (*Fig.4*)

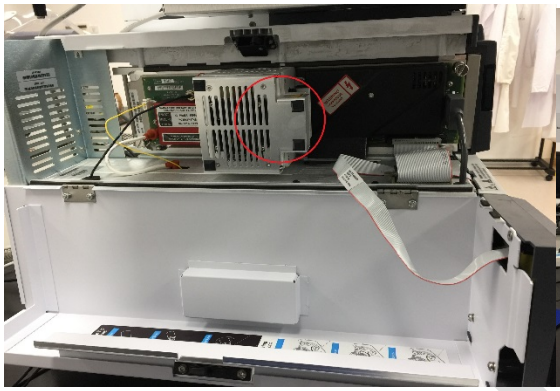


**Fig.3**

**Fig.4**

D. Turn on the PC and double click 

.When the MS temperature message show up (*Fig.5*).

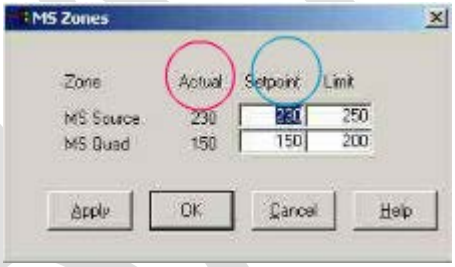
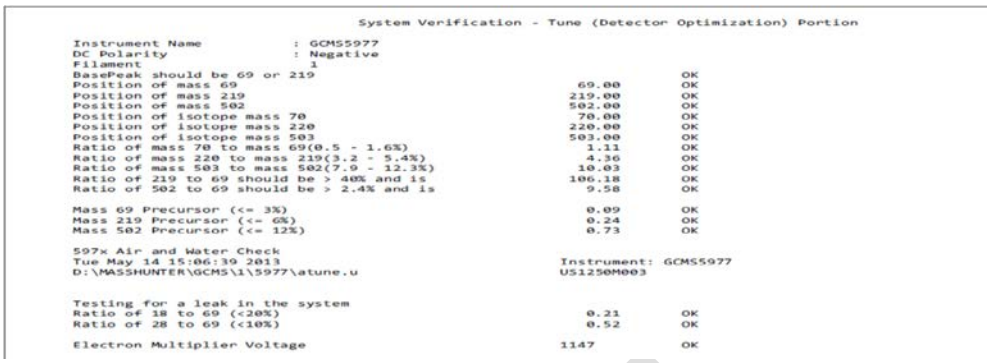



**Fig.5**

please click Apply. **DO NOT CHANGE** the temperature setting.
**Recommendation:** Leave the instrument over night before tune. If you are in a hurry, at least wait for about 2 hours until the vacuum is stable. Condition the column if necessary. The procedure of condition GC column could refer to appendix.

E. Tune: Click  to tune MS. Before samples can be analyzed using the GCMS, the mass spectrometer must be tuned, especially when GCMS has just been powered on. The MS is tuned by adjusting several parameters while the pure calibration solvent (PFTBA) is injected to the MS chamber. ATUNE.U is the chosen tune file. The tune file will be printed automatically or saved in the tune file folder.

F. Tune evaluation: Go to Tune→ Tune evaluation to check the tune result as showed in *Fig.6*. If any parameter shows fail, please check with Xiao.



System Verification - Tune (Detector Optimization) Portion

| | | |
|---|---|---|
| Instrument Name | : GCMS5977 | |
| DC Polarity | : Negative | |
| Filament | : 1 | |
| BasePeak should be 69 or 219 | | OK |
| Position of mass 69 | 69.00 | OK |
| Position of mass 219 | 219.00 | OK |
| Position of mass 502 | 502.00 | OK |
| Position of isotope mass 70 | 70.00 | OK |
| Position of isotope mass 220 | 220.00 | OK |
| Position of isotope mass 503 | 503.00 | OK |
| Ratio of mass 70 to mass 69(0.5 - 1.6%) | 1.11 | OK |
| Ratio of mass 220 to mass 219(3.2 - 5.4%) | 4.36 | OK |
| Ratio of mass 503 to mass 502(7.9 - 12.3%) | 10.03 | OK |
| Ratio of 219 to 69 should be > 40% and is | 106.18 | OK |
| Ratio of 502 to 69 should be > 2.4% and is | 9.58 | OK |
| Mass 69 Precursor (<= 3%) | 0.09 | OK |
| Mass 219 Precursor (<= 6%) | 0.24 | OK |
| Mass 502 Precursor (<= 12%) | 0.73 | OK |
| 597x Air and Water Check | | |
| Tue May 14 15:06:39 2013 | Instrument: GCMS5977 | |
| D:\MASSHUNTER\GCMS\1\5977\atune.u | US1250M003 | |
| Testing for a leak in the system | | |
| Ratio of 18 to 69 (<20%) | 0.21 | OK |
| Ratio of 28 to 69 (<10%) | 0.52 | OK |
| Electron Multiplier Voltage | 1147 | OK |

**Fig. 6**

## 5. Setting up a method

A. Open the GCMS on-line analysis software  .

B. Click “Method”, “Load Method” and open default method (*Fig.7*).


**Fig.7**

C. Save as method under user’s name.

D. Modify GC and MS parameters for the loaded method (*Fig.7*). A method is a set of parameters for the GC zone and the MS part. Basic parameters of each method mainly depend on the type of column. An application note is supplied with each column giving a typical carrier gas flow, max temperature and kind of compound it can separate.

   a. Click  to modify GC parameters.

      1) Click  to set parameters for auto-sampler.
      Choose Front Injector or Back Injector. The standard injection volume is 1 µl. Users can set the numbers and volume of solvent wash and sample wash for both PreInj and PostInj. Sample-Pumps is not recommended because it may contaminate your sample.

      2) Click  to set parameters for injection port.

In most of the case, injection port temperature is set to 250°C, but you can change this to accommodate thermal instability of your sample. Make sure that the temperature of the inlet must be hot enough to volatilize everything in your sample.

You can choose a split or splitless injection based on the concentration of your sample. If you are unsure whether the concentration of your sample is low enough to avoid overload of the mass spectrometer, it is a good idea to use split mode start with split ratio to 50:1. Splitless mode is usually for trace analysis.

Check on Gas Saver mode.

3) Click 

to set parameters for column.

Check on "Control Mode". Put the flow rate according to the application note for the column. Increase the flow rate to 2ml/min for post run.

4) Click 

to set parameters for oven.

Check on "Oven Temp On". A reasonable temperature program for an unknown is 70°C for 2 min; ramp from 70°C to the maximum temperature of the column at 15°C/min. If needed, you can use a different temperature program.

5) Click 

to set the Readiness parameters.

Select the Oven, Inlet and Detector (all attached to the column you are using now). These selections require the GCMS to wait until all setpoints related are held at a steady value before allowing a run to begin.

**Select Apply and OK when done with all sub-menus in this menu. Always remember to save your method.**

b. Click 
to modify MS parameters.

1) Set the "Solvent Delay" time (usually 3 min) to protect filament. During this time, the filament is off, so there will be no solvent peak in the chromatogram.
2) Select the scan mode: SIM or Scan.
   Full Scan mode will monitor a range of masses know as mass to charge ratio (m/z). SIM mode allows for detection of specific analysts with increased sensitivity relative to full scan mode.
3) In the Time Window, enter the run time of your method (not include the post run time)
4) Set SIM/Scan parameters.

For Scan mode, a typical mass scan range will cover from 50-550 m/z. For SIM mode, enter the masses of your interested compounds in Edit Ion Window.

5) Under 

, you can create a timed events table to decide when detector on/off according to your sample in order to increase the life of filament and multiplier.
**Select OK and save your method.**

## 6. Sample injection

A. Single injection

a. Click  for single injection.

1) Enter the **Operator name**.
2) Under "**Data Path**", always put your data under D:\DATA\<username>. You can use the browse function to select and/or create sub-directories under your username if you wish. The path must exist before you start the run.
3) Enter a **Data File Name**.
4) Enter the **Sample Name**.
5) You **MUST** specify the **Vial number** where you have already placed your sample.
6) Click on "**OK and RUN METHOD"**.

B. Sequence injection

a. Click  to edit a sequence.

1) Select your Data Path.
2) Select your method you are going to use.
3) Enter the sample name in the column with the heading "Sample" and verify the vial number.
4) Enter the method keyword and data file name.
5) Click OK.

b. Click  to test a sequence. Under this menu, if you click Run Sequence, it will not actually run this sequence. It will tell you whether there is error in your sequence or not.

c. Click  to run a sequence.

**C.** Once the run is started, a window appears that says "Override solvent delay".

**Always either ignore this message (it will go away at the end of the pre-set solvent delay) or select NO!**

## 7. Data Analysis

A. Qualitative analysis

a. Open the qualitative analysis software  .
b. Load the data you are going to analyze.
c. At this point you will see the total ion chromatogram (TIC) (*Fig.8*). You can expand an area of the TIC by holding down the left mouse button while dragging a box around the region to be expanded. To reset the display to the entire chromatogram, double-click the left mouse button.

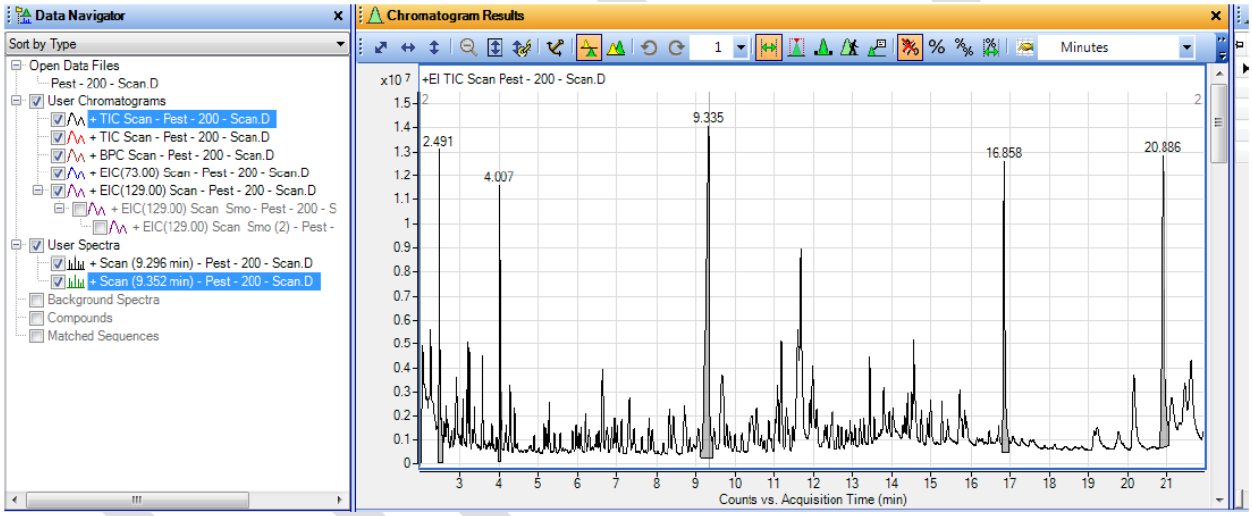



**Fig.8**

d. To view the mass spectrum: Place the mouse cursor over the appropriate retention time on TIC and double-click the right mouse button. The mass spectrum will appear in the lower window (*Fig.9*).

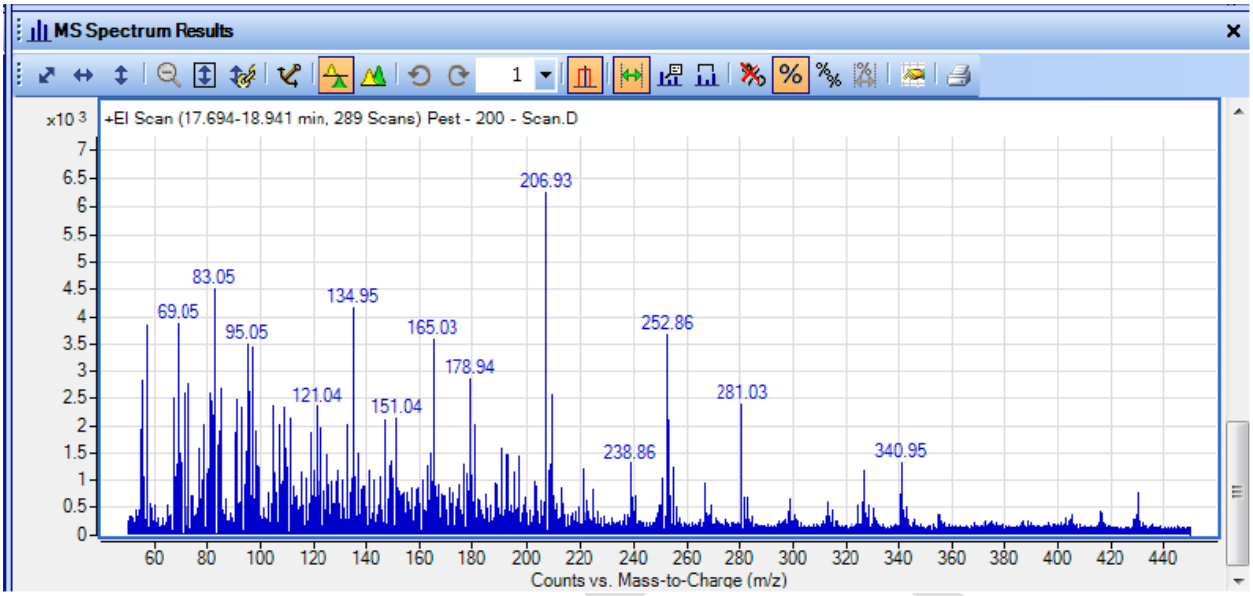



**Fig.9**

e. To perform a library search: Choose the spectra and right click. Search library for spectra. The top 10 results will show up based on possibility (*Fig.10*).

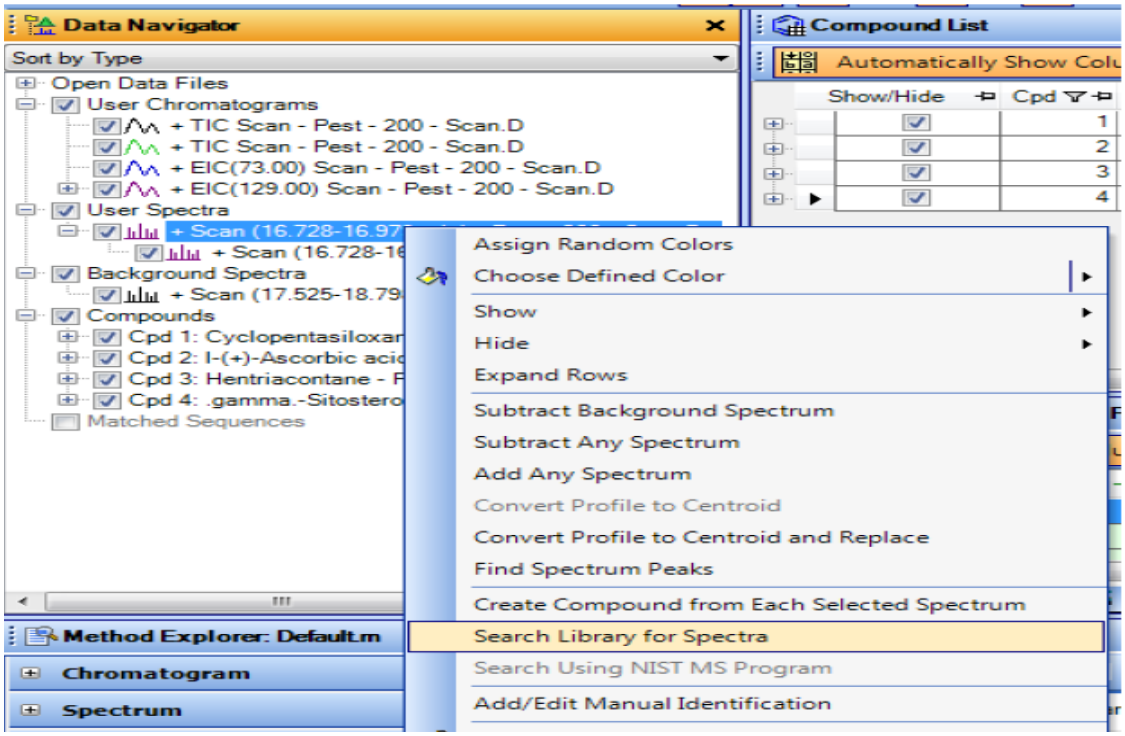



**Fig.10**

B. Quantitative Analysis

a. Open the MS Quantitative analysis software .

b. Create a new batch (*Fig.11*)

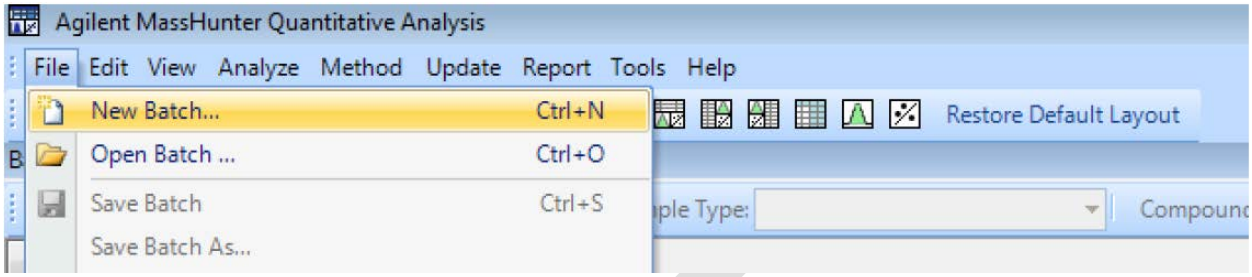



**Fig.11**

c. Add samples (*Fig.12*). Check and complete the information of sample type, level, etc.(*Fig.13*)

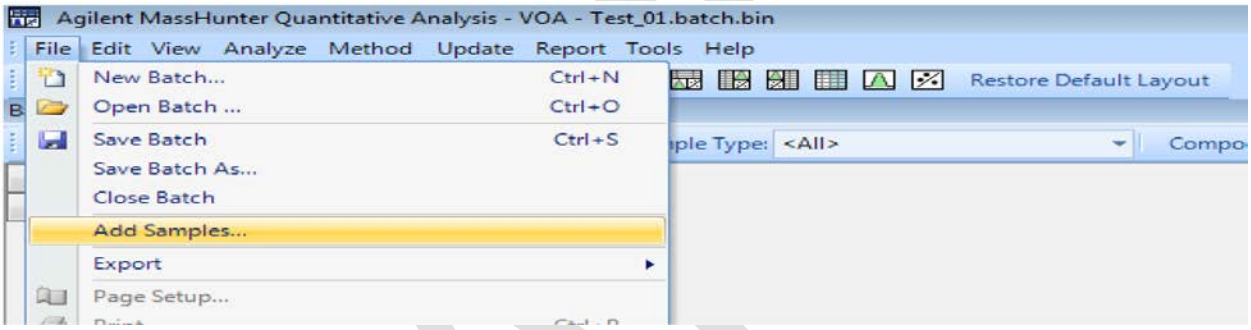



**Fig.12**

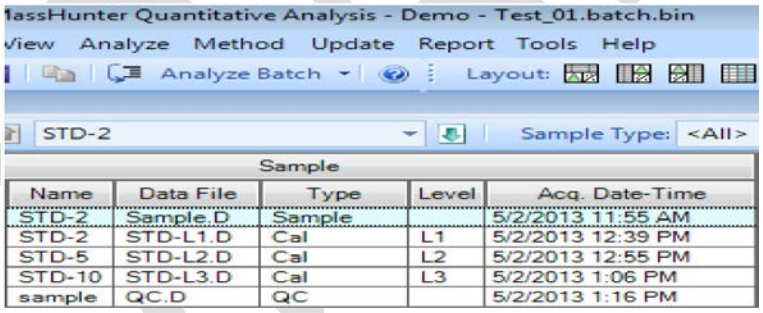



**Fig. 13**

d. Go to Method and Edit (*Fig.14*).

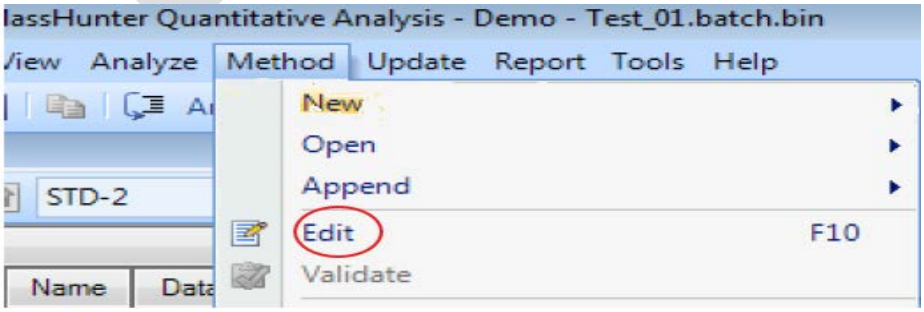



**Fig.14**

e. Compound set up. Choose the peak you are going to analyze. Double click the peak and the MS spectrum will show up. Click the quantitative ion when there is a blue triangle shows. Right click at the blue triangle and choose New compound (*Fig.15*). Choose the qualifier ion and set as New Qualifier follow the same way.

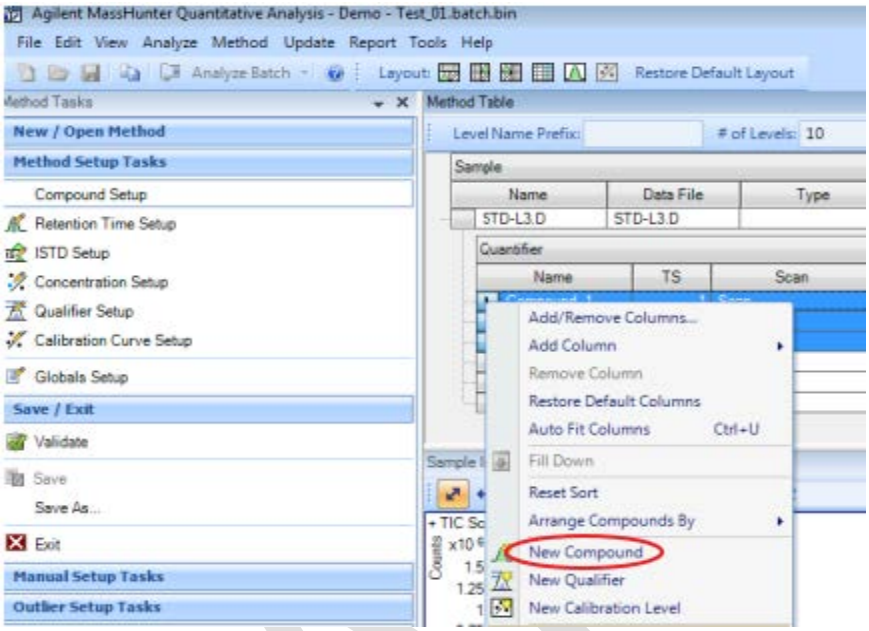



**Fig.15**

f. Click the Retention time setup and check if the value is correct (*Fig.16*).

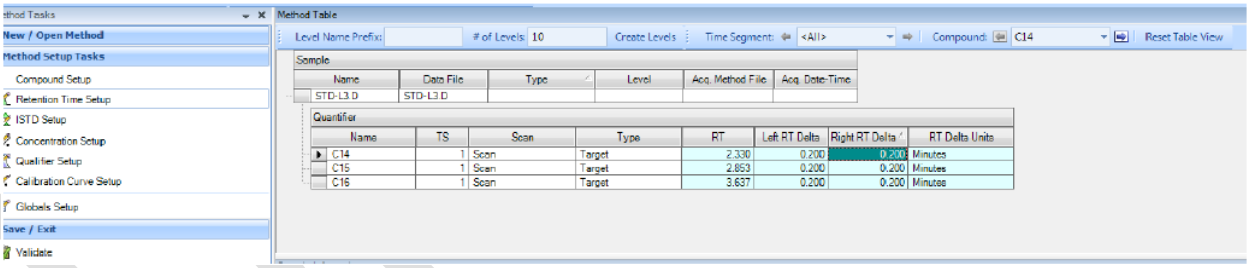



**Fig.16**

g. Concentration setup (*Fig.17*). If all the target compounds have the same concentration, go to **Method** and **Copy Calibration Levels To** other compound.

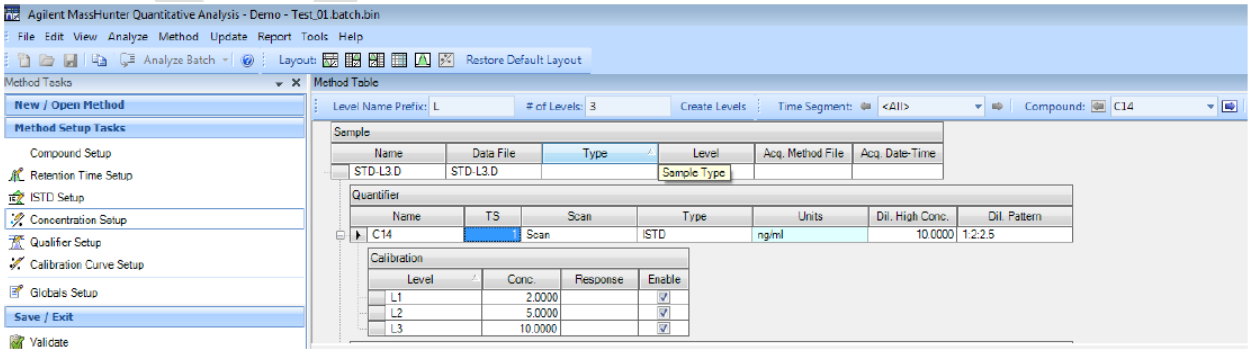



**Fig.17**

h. Calibration curve setup to choose the type of curve. And Validate method to see if there is some error (*Fig.18*).

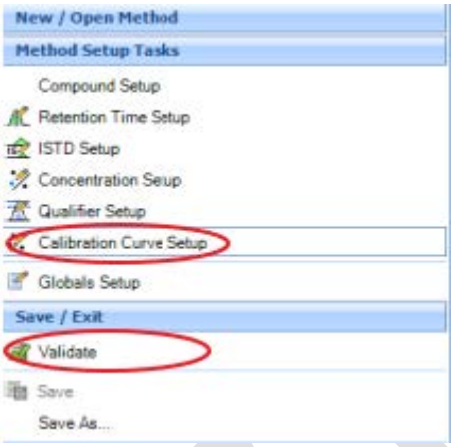



**Fig.18**

i. Click **EXIT** and **YES** to apply the quantification method to current batch**.**

## 8. Shutting down instrument.

Go to **Instrument→ MS Vacuum control→ Vent.** You could monitor the Turbo pump speed and MS temp (*Fig.19*). When all the criteria are meet, you could power off the instrument.

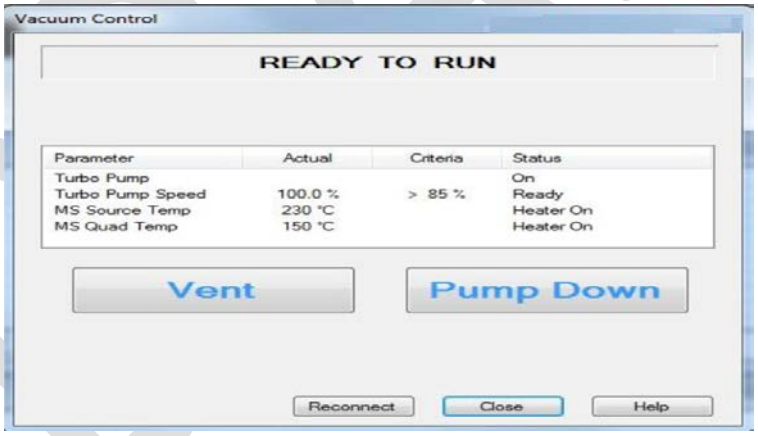



**Fig. 19**

## 9. Cleaning

After analyzing, users need to clean their sample vials from the sample tray.

## 10. Appendix

**How to condition a capillary column**

1. Install the column in the GC inlet.
2. Set a minimum velocity of 30cm/s or as recommended by the column manufacturer. Allow the carrier gas to flow through the column at room temperature for 15-30min to remove air.
3. Program the oven from room temperature to the maximum temperature limit for the column.
4. Increase the temperature at a rate of 10-15 °C/min
5. Hold at the max temperature for 30mins.
6. Set the GC oven temperature to 30 °C and wait for the GC to become ready.
7. Attached the column to the interface.

## 11. Reference

https://wenku.baidu.com/view/f8a53bd6c5da50e2534d7fbe.html

## Document information:

| | | | |
|---|---|---|---|
| Document NO. | GOP-GCMS-01 | Version NO. | 0.1 |
| Drafted/Modified by: | Xiao Zhou | Effective Date: | |
| Comments: | | | |