Operating Instructions for the Shimadzu GCMS QP-2020 NX Gas Chromatograph Mass Spectrometer

Research Instrumentation Center – BRWN 3154
Revised on Nov. 23, 2021 by R. Hilger

Introduction

The Shimadzu QP-2020 NX GC-MS system consists of an autosampler, a gas chromatograph (GC), and a mass spectrometer (MS). The autosampler is capable of processing up to 150 samples in an automated manner. The GC consists of a sample inlet, a column, and systems for controlling the temperatures of these components as well as the flow of helium carrier gas. The MS is an electron ionization single quadrupole instrument.

Sample solution is injected into the GC inlet where it is vaporized, entrained in the carrier gas, and applied to the top of the column. The analyte then proceeds through the column at a rate determined by its interactions with the stationary phase and mobile phase. Typically, elution time increases with the boiling point of the analyte. The temperature of the column is typically increased using a linear ramp over the course of the analysis time in order to separate a wide range of analytes in the shortest possible time. The eluate from the GC column is introduced into the MS where it is bombarded by electrons in order to generate positive ions. The ionization process also causes fragmentation of most analytes. The mass-to-charge ratios of the analyte ions and fragment ions are measured using a quadrupole mass filter. The mass spectra may be searched against a database of spectra acquired under similar conditions in order to identify the analyte.

Appropriate Samples and Sample Prep

Generally speaking, only volatile compounds should be injected onto the GC-MS. Components of your sample with boiling points beyond the temperature range accessible by the GC-MS (about 350 °C) will become permanently adsorbed to the GC column, reducing its performance and eventually destroying it. **GC-MS samples generally should not contain strong acids, strong bases, polymers, detergents, or nonvolatile salts.** Also, it is wise to ensure that your samples are in a GC-appropriate solvent. The solvent should have a lower boiling point than any of your analytes so that the solvent is first to elute from the GC column. Common GC solvents include methanol, hexane, acetone, ethyl ether, and dichloromethane. Water is generally not considered a good GC solvent due to its high expansion volume. Additionally, water can damage certain types of GC columns. **Samples containing significant amounts of water should not be injected onto the GC-MS without consulting RIC staff.**

The autosampler only accepts samples in a very specific vial with a very specific cap and septum. Septa should be placed on the vials with the red side down. **Samples in improper containers may not be correctly processed by the autosampler and may damage the autoinjector.** Users are generally advised to purchase their own vials, caps, and septa using the part numbers listed below. The vials should be at least half full of sample to ensure aspiration of sample by the syringe needle. There are inserts available that allow analysis of sample volumes down to 250 µL.

Vendor	Part Number	Description
VWR	66030-920	Amber glass vial, 2 mL
VWR	66030-934	Screw thread cap with septum
VWR	66030-902	400 uL insert for low volume analysis
VWR	66030-380	Clear glass vial, 4 mL
VWR	66030-392	Black cap 13-425
VWR	66030-388	Teflon septum

GCMS Real Time Analysis Software

When you first start up the GCMS Real Time Analysis software, the screen should look something like Figure 1. The Assistant Bar on the left hand side provides access to the menus that control the instrument's functions.
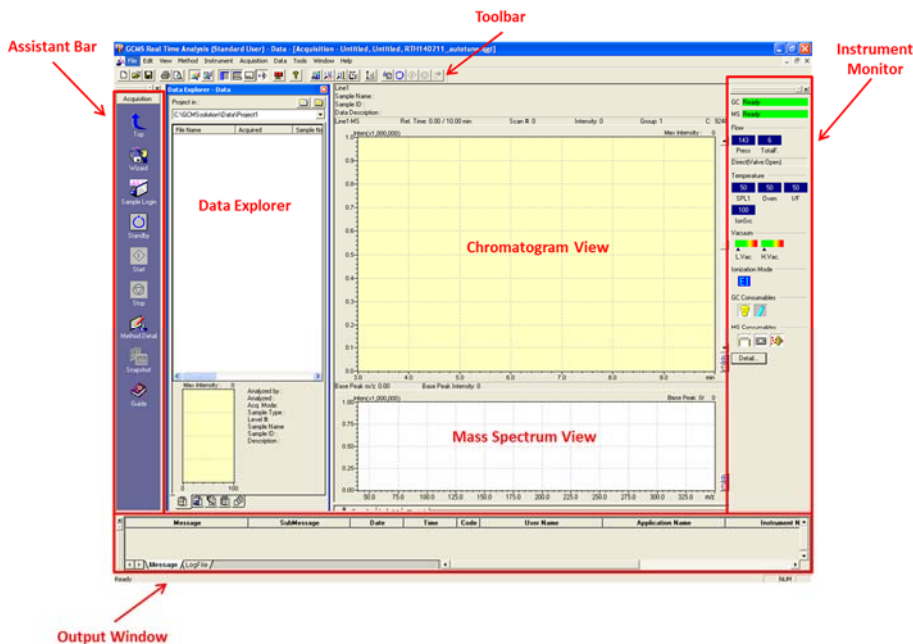
Figure 1: GCMS Real Time Analysis software window highlighting the various panes.

- The Top button ![Top] may be used to back out of the presently selected menu. Clicking the top button on the Assistant Bar will reveal the instrument's various menus.

- The Data Acquisition menu ![Data Acquisition] is used to set up the instrument for analysis.

- The Data Analysis menu ![Data Analysis] is used to analyze and process previously acquired data.

- The Batch Processing menu ![Batch Processing] is used to set up your sample run.

- The System Configuration menu ![System Configuration] allows you to view the current configuration (e.g. injector type, column type) of the instrument.

- The Tuning menu ![Tuning] is used to tune and calibrate the MS.

Certain functions can also be accessed via the Toolbar along the top of the window. Mouseover the various icons on the Toolbar to examine their functions. The Data Explorer pane allows easy access to the files associated with the current project. The tab controls at the bottom of the pane display files of different types (e.g. method files, batch files, data files). The project folder can be changed by accessing **File > Select Project(Folder)**. The Output Window displays any messages (typically errors) generated while the instrument is in operation. The Instrument

Monitor pane displays the status of the instrument's various components including the GC inlet pressure in kPa (Press), the carrier gas total flow rate in mL/min (Total F.), and the current state of the split valve (Close vs. Open). The Instrument Monitor also displays the temperatures (in °C) of the GC inlet (SPL1), the column oven (Oven), the MS interface (I/F), and the MS ion source (IonSrc). The status of the MS vacuum system is also displayed along with the ionization mode. The status of various consumables on the instrument is also displayed; mouseover each one to examine its remaining lifetime. The View menu can be used to add/remove the various panes from the window.

Tuning the Mass Spectrometer

It is a good idea to tune the MS at the beginning of each day. Use the following procedure to tune the MS:

The instrument is now equipped with an ion source switchable between EI and CI. In the tuning window, make sure you tuned the mode you want to use for your analysis.

1. Access the Tuning menu from the Assistant Bar.

2. Click on **File > New Tuning File**. For tuning mode, select **Normal**

3. Click on Start Auto Tuning in the Assistant bar. Wait for auto tuning to complete (about 3 min). The Tuning Result View should automatically appear upon completion of auto tuning (see Figure 2 and Figure 3). If this fails to occur, it can be accessed via the Assistant Bar.

4. Verify that the FWHM of each peak in the Peak Profile (Figure 2) tab is in the range 0.5 - 0.7. Verify that the detector voltage does not exceed 2 kV. Verify that either m/z 69 or m/z 219 is the base peak (Ratio = 100.00). Verify that the relative intensity ratio for m/z 502 is at least 2%.
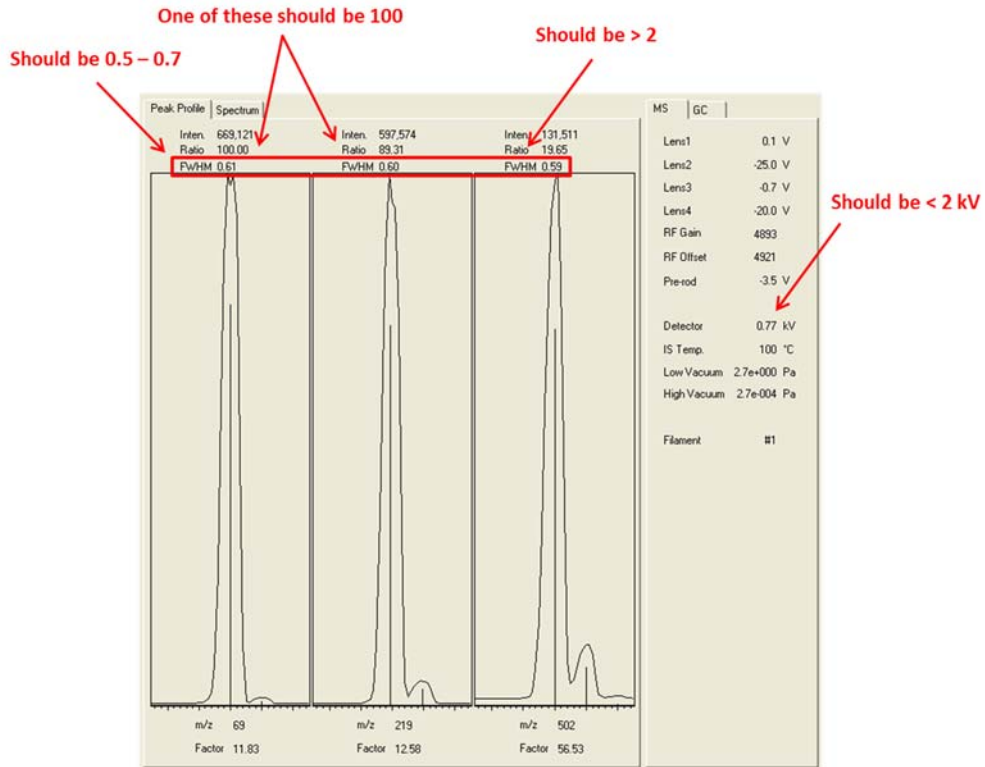
Figure 2: Peak profiles in Tuning Result View

5. On the Spectrum tab (Figure 3), verify that the intensity for m/z 69 is at least twice that for m/z 18. If any of these conditions are not met, the instrument is not operating properly and should not be used.
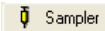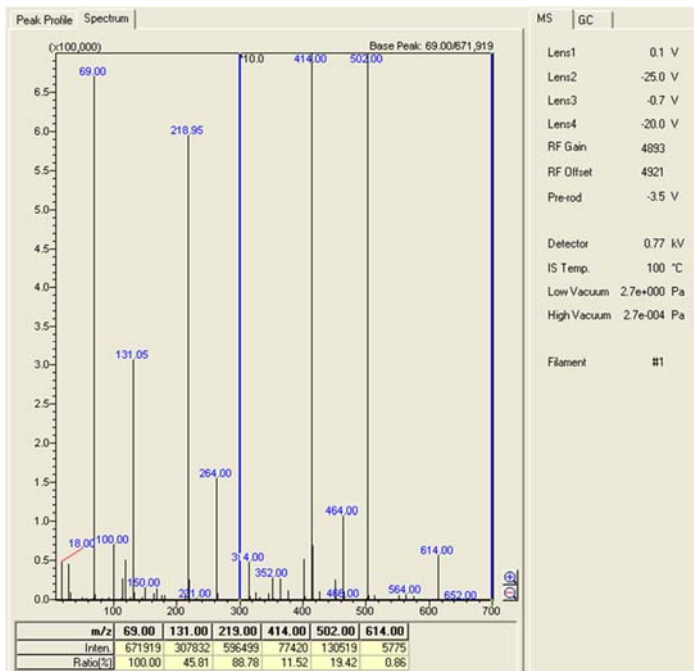

Figure 3: Spectrum in Tuning Result View

6. Click **File > Save Tuning File As**, and save the tuning file in your project folder.

Creating a Method File

The method file contains all of the parameters that the autosampler, GC, and MS will use to analyze your samples. Use the following procedure to define these parameters prior to starting your analysis:

1. Click on Data Acquisition ![Data Acquisition] then Method Detail ![Method Detail] using the Assistant Bar. If you need a starting point for your method, click on **File > Open Method File**, and select **C:\GCMSsolution\Data\New_User\GCMS_default_method.qgm**.

2. The autosampler settings are displayed on the sampler tab ![Sampler]. The only settings that might require adjustment are the rinse settings. You can alter the number of times that the syringe is rinsed with solvent before and after each injection, as well as the number of rinses with sample prior to injection.

3. Setting up the GC on the GC ![GC] tab:

   a. SPL1 ![SPL1] contains the settings for the inlet.

      i. **Temperature** sets the temperature of the inlet, which should be higher than the boiling point of your highest boiling sample components. For samples that may contain high-boiling compounds, a high temperature of **250 °C or above** is suggested for your initial run.

      ii. **Injection Mode** should be set to **Split** for typical analyses.

      iii. The settings below **Flow Control Mode** determine the various flow rates of helium gas throughout the GC. These settings depend on each other to some degree, so when you change one of these settings, others may be automatically adjusted.

         1. The most important of these settings is the **Column Flow**, which is typically 0.5 - 2.0 mL/min for narrow bore capillary columns. **Linear velocity** is basically the column flow expressed in different units.

         2. The **Pressure** is the value required to push the carrier gas through the system.

3. **Total flow** is the sum of all carrier gas flows.

4. The **Purge Flow** is typically maintained at **3 mL/min**.

5. The **Split Ratio** determines what fraction of the injected sample that is applied to the column. For example, a split ratio of 80 means that for every 50 units of gaseous sample that are thrown away to waste, 1 unit goes into the column. A split ratio of **100** is a good place to start. The split ratio may be decreased if the analyte is low abundance (signal-to-noise ratio is poor), or increased if the signal is too intense.

b. Column ⟨ Column ⟩ contains the temperature settings for the column.

   i. **Temperature** sets the initial temperature of the column oven. This should be low enough to allow for adequate retention of your lowest boiling analytes. Typical values range from **30 - 60 °C**.

   ii. **Equilibration Time** between when the column reaches the initial temperature and when the sample is injected, typically **3.0 min**.

   iii. The **Column Oven Temperature Program** controls how the temperature of the column is ramped over the course of the analysis.

      1. **Rate** controls the rate of temperature increase in °C/min. Lower Rate values may improve the resolution of the separation at the cost of longer analysis time.

      2. **Temperature** is the final temperature of the method segment.

      3. The **Hold Time** controls the time (in min) to hold the column at the final temperature before starting the next segment.

      4. The final temperature of your analysis should be high enough to elute everything in your sample so that nothing is left to contaminate subsequent analyses. For samples that may contain high-boiling compounds, a high final temperature such as **250 °C or above** is advisable for your initial run.

4. Setting up the MS on the MS ⟨ MS ⟩ tab:
   The instrument is now equipped with an ion source switchable between EI and CI.

In the MS tab, make sure you choose the mode you want to use for your analysis.

a. The **Ion Source Temp.** and the **Interface Temp.** should both be set to at least the highest temperature reached by the column. It is typical to set them to the same value as the inlet temperature.

b. The **Solvent Cut Time** sets the time delay between sample injection into the GC and the activation of the ionization filament in the MS. This delay should be sufficient for the solvent to elute from the GC column. Ionizing the large quantity of eluted solvent reduces the lifetime of the filament and dirties the ion source. A value of 1.5 min is a good guess for most GC solvents. You will need to increase this time if you observe the large solvent peak in your chromatogram. You may need to slowly reduce this time if you have low boiling analytes that elute near the solvent peak.

c. The **Start Times** and **End Times** determine the time window over which the MS is active after injecting the sample into the GC. The **start time cannot be less than the Solvent Cut Time** and the **end time cannot be longer than the Total Program Time shown on the column section** 🔴 Column **of the GC** 🔴 GC **tab**.

d. The **Acquisition Mode** can be set to either **Scan** which scans the MS over a specified range of m/z, or **SIM** which stands for single ion monitoring. In SIM mode the MS monitors signals only at specified values of m/z.

e. The data acquisition rate is determined by the **Event Time**, which should be kept to 0.5 s or less in order to ensure a sufficient number of points across each GC peak.

f. The **Scan Speed** in m/z per sec is calculated by dividing the m/z range by the Event Time.

5. Once the parameters for the autosampler, GC, and MS have been defined, click **File > Save Method File As**, and save the method in your project folder.

Creating a Batch File

The batch file lists all of the samples that you want to analyze during the run and specifies the parameters for the instrument to use while analyzing each sample. The batch file also specifies each sample's location in the sample rack. The autosampler can be used to automatically process up to 150 samples. The autosampler plucks the sample to be analyzed from the sample rack (Figure 4) and places it in the autoinjector's tray (Figure 5). After the

sample is injected, the vial is replaced in the sample rack. Use the following procedure to create a batch file:

1. In the Assistant Bar, click on the Batch Processing ![Batch Processing] menu then settings ![Settings] . In the window that pops up, click the **Folder** tab and specify your project folder. Click OK.

2. Click the Wizard button ![Wizard] on the Assistant Bar. The wizard will take you through the process of creating a batch table.

   a. You can either create a new table, or append samples to an existing table.

   b. For the **Sample Type** select **Unknown Only**

   c. Specify a method file for the analysis.

   d. Uncheck the boxes under **Data Processing** and click **Next**

   e. **Vial #** specifies the location of the first vial in the sample rack (see Figure 4). The vials are numbered 1 – 150 with position 1 being the first position in rack 1 and position 150 being the last position (position 25) in rack 6. Your vials must be placed consecutively starting from the specified location. If the instrument fails to find a vial in the specified location, data acquisition will stop.
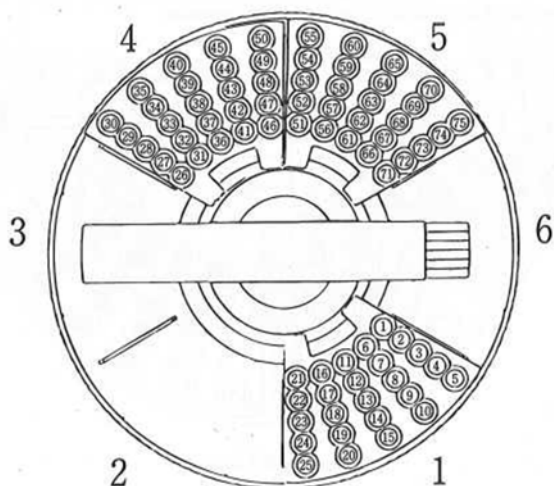

Figure 4: Diagram of the sample tray showing the location of each rack as well as the location of each sample within each rack.
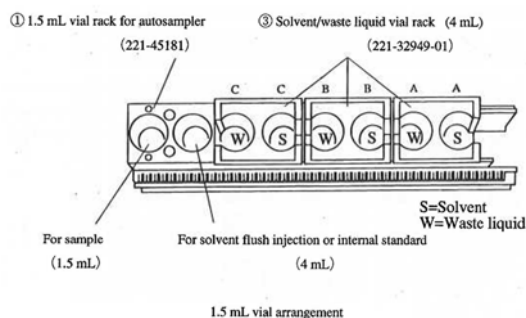

Figure 5: Diagram of the autoinjector tray.

   f. **Sample Count** is the total number of samples in your run

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g. The typical **Injection Volume** is 1 uL.

h. Optionally fill out the **Sample Name** and **Sample ID**. If the **Auto-increment** box is checked, a number corresponding to the sample's place in the sequence is automatically added to the sample ID.

i. Click the **Next** button and enter a **Data File Name** which can also be auto-incremented.

j. Uncheck the **Report Out** box unless you want to set up the reporting function (see manual for details) and enter an optional **Data Description** and click **Finish.**

3. After your batch table appears you can edit it directly if desired.

4. **Click File > Save Batch File As** and save the batch file in your project folder.

Performing a Run

Before starting the run, make sure that your samples are loaded into their proper places in the sample rack (see Figure 4). **Ensure that the solvent vial is in place (Solvent C in Figure 5) and contains an adequate amount of solvent.** Ensure that a waste bottle is in place (Waste liquid C in Figure 5) and that it is not in danger of overflowing. Click Start [▶ Start] on the Batch Processing [Batch Processing] menu of the Assistant Bar to begin automated analysis. After the method is downloaded to the instrument, it may take a few minutes for the temperatures of the various zones to be established. The Instrument Monitor pane will show the current temperatures along with the setpoints. Once both the GC and MS show **Ready** on the Instrument Monitor, an equilibration time of a few minutes will pass prior to injection of the first sample. The Pause/Restart [❚❚ Pause/Restart] button can be used to pause batch processing at any time. Batch table rows that have yet to be processed can be edited during the pause. Batch processing can be aborted by pressing the Stop [■ Stop] button. The chromatogram and mass spectra can be viewed on the data acquisition menu [Data Acquisition] while the run is in progress. If you do not see the trace on the screen, you may need to unzoom by right clicking the plot and selecting **Initialize Zoom**. You can also

right click the plot and select **Display Settings** to adjust the y-axes.

Analyzing and Exporting Data

Data can be analyzed and exported to ASCII (text) files using the GCMS Postrun Analysis

program, which can be accessed from the desktop or by pressing Data Analysis [Data Analysis icon] on the Assistant Bar. Below is an example of a typical layout in the postrun analysis software:
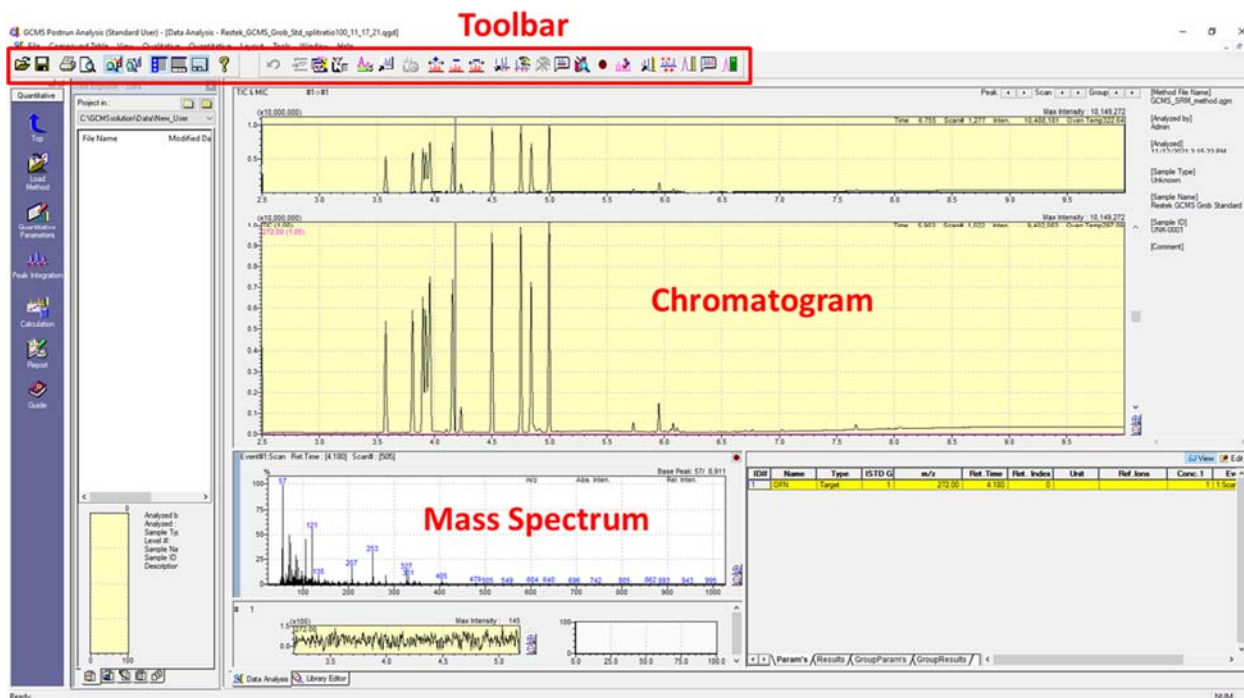
1. Saving a chromatogram as an ASCII (text) file suitable for Excel:

   a. Open the file in GCMS Postrun Analysis

   b. Click **File → Export Data**

   c. Select **Output File** and enter a file name

   d. Check the box next to **Chromatogram (TIC/MIC)** and click **OK**

2. Viewing mass spectra associated with peaks in the chromatogram:

a. Click the **Manual Peak Integrate** button ⬛ on the toolbar then left click and drag across a peak in the chromatogram. Repeat this process for all peaks of interest in the chromatogram.

b. Step through your peaks using the arrows at the top right Peak ◀ ▶ to view the mass spectra associated with each peak.

c. For each peak that you wish to save a mass spectrum, right click the mass spectrum and select **Register to Spectrum Process Table**

3. Saving mass spectra from a data file as an ASCII (text) file suitable for Excel:

a. When you have completed all the steps in 2 above, click **File → Export Data**

b. Select **Output File** and enter a file name

c. Check the box next to **Spectrum** and click **OK**. The spectra for all peaks will be saved in the same text file.

4. Searching mass spectra against a library in order to identify compounds based on their fragmentation patterns:

a. Perform the steps in 2 above.

b. View the mass spectrum of the desired peak using the arrows at the top right Peak ◀ ▶

c. Right click on the mass spectrum and select **Similarity Search**. Your mass spectrum will be displayed along with the closest matches from the library.

d. In the results window click **Report → Search Results** to generate a pdf of the results.

5. Integrating peaks in the chromatogram:

a. Perform the steps in 2 above

b. Right click on the chromatogram and select **Show Qualitative Table**

c. To export the data in the table, drag over it, copy it, then paste it into a notepad file.

Shutting Down the GC-MS

The instrument should be left in a standby state when not in use to minimize both unnecessary consumption of carrier gas and unnecessary wear on the instrument. When you are finished using the instrument, use the following procedure to shut down the instrument.

1. Click on Data Acquisition on the Assistant Bar.

2. Click **File > Open Method File** and open the system idle method e.g.
    C:\GCMSsolution\Data\New_User\system_idle.qgm.

3. Click Download then click OK on the two prompts to download the method to the instrument.

4. After the method is downloaded, click the Stop button and confirm that you want to stop acquisition.

5. Verify that the total flow of carrier gas is 5 mL/min using the Instrument Monitor. You may now close the GCMS software and log out of the computer.

Contacts

If you notice anything wrong with the instrument or you encounter any problems while using it, please follow the shutdown procedure above and contact the people listed below:

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