# Operating Instructions for the Shimadzu GC-2010 Gas Chromatograph

Research Instrumentation Center – BRWN 3154
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## Introduction

The Shimadzu GC-2010 gas chromatography system consists of an autosampler and a gas chromatograph (GC) equipped with a flame ionization detector (FID). 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 FID detects eluting compounds by ionizing them in a hydrogen flame and detecting the positive ions as a current. This detector is particularly well suited to organic compounds, although sensitivity may be reduced for compounds containing many highly electronegative atoms such as oxygen, fluorine, and nitrogen.

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 FID where it is burned in a hydrogen flame in order to generate positive ions. The positive ion current generates the signal.

## Appropriate Samples and Sample Prep

Generally speaking, only volatile compounds should be injected onto the GC. Components of your sample with boiling points beyond the temperature range accessible by the GC (about 300 °C) will become permanently adsorbed to the GC column, reducing its performance and eventually destroying it. **GC 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 without consulting AAIC 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.** AAIC has a very limited quantity of vials that can be made available to users with a limited number of samples, but 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 |

## Starting Up the GC System

1. Open the valves on the hydrogen and air cylinders located at the end of the bench (only the valve on top of each cylinder). Make sure each valve is open at least a few turns.

2. Log in to the computer and start the GCsolution software. Click the **Operation** tab and you will see the little window shown in Figure 1. Click the little GC with the number 1 next to it and log in as a Standard User (no password).


**Figure 1**

3. Once the software opens click on **File → Select Project(Folder)** and select your project folder (create a folder if you do not have one).

## GCsolution Software Overview

The Assistant Bar (Figure 2) appears on the left side. It is used to access the various menus and commands that control the instrument. The **Instrument Parameters** menu 

is used to configure the GC for analysis. The **Download Parameters** button 

downloads the setup to the instrument. The **Batch Processing** menu 

is used to setup a spreadsheet called a batch table that contains information about each sample that you wish to process during the automated run. Some commands are also available via the toolbar that runs along the top of the window. The Instrument Monitor (Figure 3) can be toggled on and off using the  button on the toolbar. The Instrument Monitor provides information on the instrument's various components including status, temperatures, and flow rates. The Data Explorer window (Figure 4) can be toggled on/off using the  button on the toolbar. The tabs at the bottom of the Data Explorer window provide quick access to the various files (data files, method files, batch files) in your project folder.


Figure 2

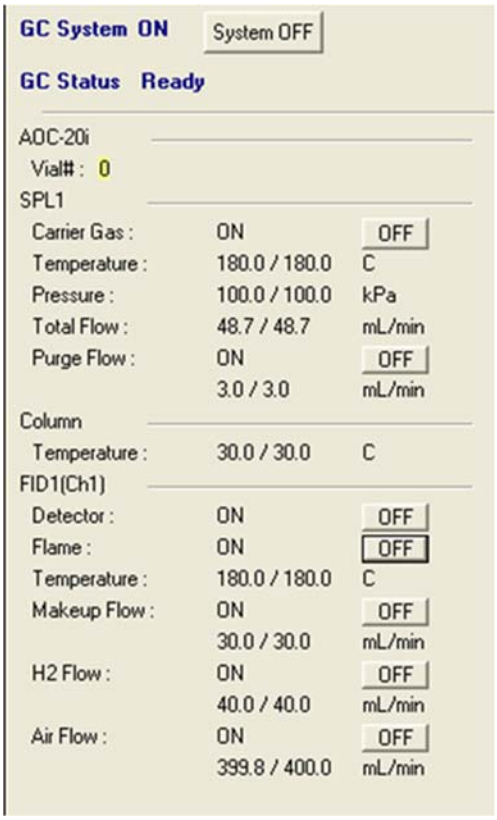



Figure 3


Figure 4

## Setting up the GC System (Creating a Method File)

1. Click the **Instrument Parameters** button 

on the Assistant Bar. If you need a starting point for your method, click on **File → Open Method File**, and select **C:\GCsolution\Data\RTH\TolBenzDMF.gcm**.

2. Click the autosampler tab 

to define settings for the autosampler. Under most circumstances, an injection volume of 1 µL is appropriate. You can also choose the number of times the syringe is rinsed with solvent before and after injecting each sample, as well as the number of times the syringe is rinsed with the sample itself prior to injection.

3. Set up the injection port by clicking the 

tab.

    a. The injection port temperature should be higher than the boiling point of your highest boiling sample components. For samples that may contain high-boiling compounds, a high injection port temperature of 250 °C or above is suggested for your initial run.

b. Injection Mode should be set to *Split*.

c. Flow Control Mode must be set to *Linear Velocity*.

d. Purge Flow should be 3 ml/min.

e. Linear Velocity should be adjusted such that Column Flow is in the range 0.5 – 1 ml/min.

f. Split Ratio = 80 is a good starting point for most samples. With this setting, 1 part in 80 injected will be applied to the column. It is best to start with a high split ratio to avoid overloading the column. If the resulting signal-to-noise is poor, the split ratio can be decreased to boost signal intensity.

4. Set up the column oven by clicking the 

tab.

a. A typical equilibration time is 3 min.

b. The column oven temperature program controls how the temperature of the column is ramped over the course of the analysis.

i. The column oven temp. 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. 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.

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

iv. 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 is advisable for your initial run.

5. Set up the FID by clicking the 

tab.

a. The detector temperature must be hot enough to maintain each of your analytes in the gas phase; it also must be greater than or equal to the final temperature of the column

oven.

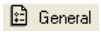
b. A typical sampling rate is 40 ms. Higher sampling rate increases signal but provides fewer points across each peak.

c. Delay time is fixed at 0 min.

d. Makeup Flow should be set to 30 ml/min.

e. H2 Flow should be set to 40 ml/min.

f. Air Flow should be set to 400 ml/min.

6. The General tab 

typically does not require modification.

7. On the Assistant Bar click the **Download Parameters** button 

to download the settings to the instrument.

8. Turn on the Instrument Monitor using the  button on the toolbar. You should see the temperatures changing to the set points you defined earlier.

9. Using the Instrument Monitor, turn the detector on.

10. Using the Instrument Monitor, turn the flame on. The air and H2 flows should turn on. Wait for 1 min to see if ignition is successful (Flame changes to *On*). Sometimes the ignition fails which is characterized by persistent fluctuation of the air flow. If this occurs simply toggle the flame off, wait for the air flow to stabilize at 400 ml/min, then toggle the flame back on.

11. Once the GC Status changes to *Ready* on the Instrument Monitor, in the chromatogram pane click on **Zero Adjust**.

12. In the chromatogram pane click on **Slope Test**. Click OK once the result is displayed.

13. Save your method file in your project folder by clicking **File → Save Method File As**

## 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 5) and places it in the autoinjector's tray (Figure 6). After the sample is injected, the vial is replaced in the sample rack. Use the following procedure to create a batch file:

1. On the Assistant Bar click **Batch Processing** 

then **Settings** 

. Select the folder tab, click **Use Specified Folder** and select your project folder. This ensures that your data files will be saved in your project folder.

2. On the Assistant Bar click **Batch Table Wizard** 

to begin creating your batch table.

    a. Specify *new* to start from scratch or *append* to add entries to the current batch table.

    b. Specify your method file then click **Next**.

    c. Specify 1 group, *Unknown Only*, click **Next**.

    d. Enter the number of samples you want to run as the Sample Count as well as the number of Repetitions.

    e. Enter the location of your first sample in the sample rack as the Vial #. Your samples must be placed consecutively in the sample rack.

    f. You can enter a Sample Name and Sample ID. The Auto Increment option will automatically append consecutive numbers to your sample names and ID's during the run.

    g. Specify lSTD Amount = 1 then click **Next**.

    h. Enter a Data File Name, specify no Report Out, enter an optional Data Description, then click **Next.**

i. Specify no Summary Report then click **Finish**.

3. Your batch table will be displayed. You can edit the batch table directly if desired.

4. Save your batch table in your project folder by clicking **File → Save Batch File As**.

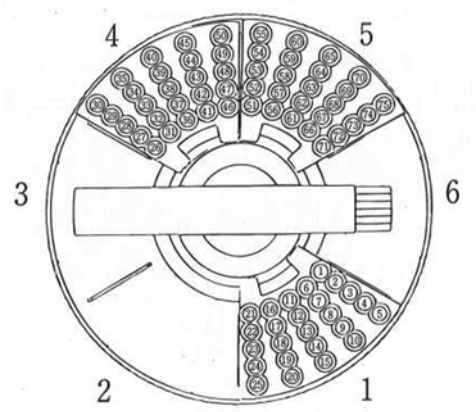



**Figure 5:** Diagram of the sample tray showing the location of each rack as well as the location of each sample within each rack.

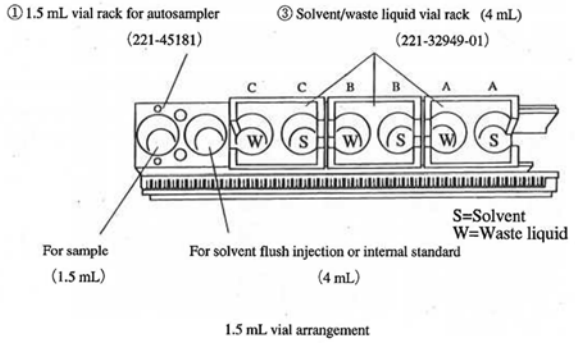



**Figure 6:** Diagram of the autoinjector tray.

## Performing a Run

Before starting the run, make sure that your standards and unknowns are loaded into their proper places in the sample rack (see Figure 5). Ensure that the solvent vial is in place (Solvent A in Figure 6) and contains an adequate amount of solvent. Ensure that waste bottles are in place (Waste liquid A and Waste liquid B in Figure 6) and that it is not in danger of overflowing. Click **Start** 

on the **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 the GC Status shows *Ready* on the Instrument Monitor, an equilibration time of a few minutes will pass prior to injection of the first sample. The **Pause** 

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** 

button. The chromatogram can be viewed 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 **Undo Zoom**. You can also right click the plot and select **Chromatogram Display Settings** to adjust the y-axis.

## Data Analysis

To analyze your data files make sure the Data Explorer pane is visible by clicking the button on the toolbar. In the Data Explorer pane, select the data files tab to see the data files in your project folder. Double click on any data file to open it for analysis.

1. Integrating Peaks

    a. The GC Postrun software should automatically find and integrate peaks in each data file that is opened. The peaks should be denoted in the chromatogram with little red arrows. If you don't see the peak table with the height and area values, click **Switch Layout** below the menu bar.

    b. If you want to add or remove peaks from the table, click **Manual Peak Integration** 

on the Assistant Bar. You can use the tools in the *Manual Peak Integration* window to add, remove, or alter the detected peaks. For example, to add a peak you can click **Insert Peak** then click in the chromatogram on the two boundaries of the peak you want to add.

2. Exporting Data

    a. To save the chromatogram as an ASCII (text) file suitable for Excel, back in the data acquisition software, right click the data file in the Data Explorer pane and click **File Convert**, then select **Data file to ASCII file**, select your project folder and click **Ok**.

    b. To export the data in the peak table, in GC Postrun, click **View → Peak Table → Copy Whole Table**, then paste the data into Notepad and save.

## Shutting Down the GC

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. Using the Instrument Monitor, turn the flame off then turn the detector off.

2. Close the valves on the air and hydrogen cylinders (just one valve on top of each cylinder).

3. On the Assistant Bar click **Instrument Parameters** 

. Click **File → Open Method File → C:\GCsolution\Data\RTH\system_idle.gcm**

4. On the Assistant Bar click **Download Parameters** 


5. Using the Instrument Monitor, verify that the total flow of carrier gas is 0.5 - 1 mL/min and the temps have been set to 50 °C.

6. You may now close all software windows and log out of the computer.