

<b>UM</b>	<b>JABATAN KIMIA</b>	<b>Kategori</b>	<b>Arahan Kerja</b>
	<b>PROSEDUR PENGENDALIAN PERALATAN TGA-GCMS BAGI ANALISIS SAMPEL</b>	<b>Dokumen No.</b>	<b>AK/JK.P/F/01</b>
		<b>Tarikh   Semakan</b>	<b>09.08.2018   1</b>
		<b>Mukasurat:</b>	<b>1 daripada 7</b>

### 1. TUJUAN

Tujuan prosedur ini dtubuhkan adalah untuk memberikan arahan yang jelas bagi langkah atau tatacara bagaimana menjalankan analisis sampel menggunakan peralatan Thermogravimetry-Chromatography Mass Spectrometry (TGA-GCMS) for Pyris 1 TGA.

### 2. SKOP

Terpakai untuk pengguna dari Jabatan Kimia.

### 3. PROSEDUR ARAHAN

#### 1) Always switch on Pyris 1 TGA system in “CAT” sequence :-

- i) C- Computer.
- ii) A- Analyzer.
- iii) T- TAGS( Themal Analyzer Gas Station).

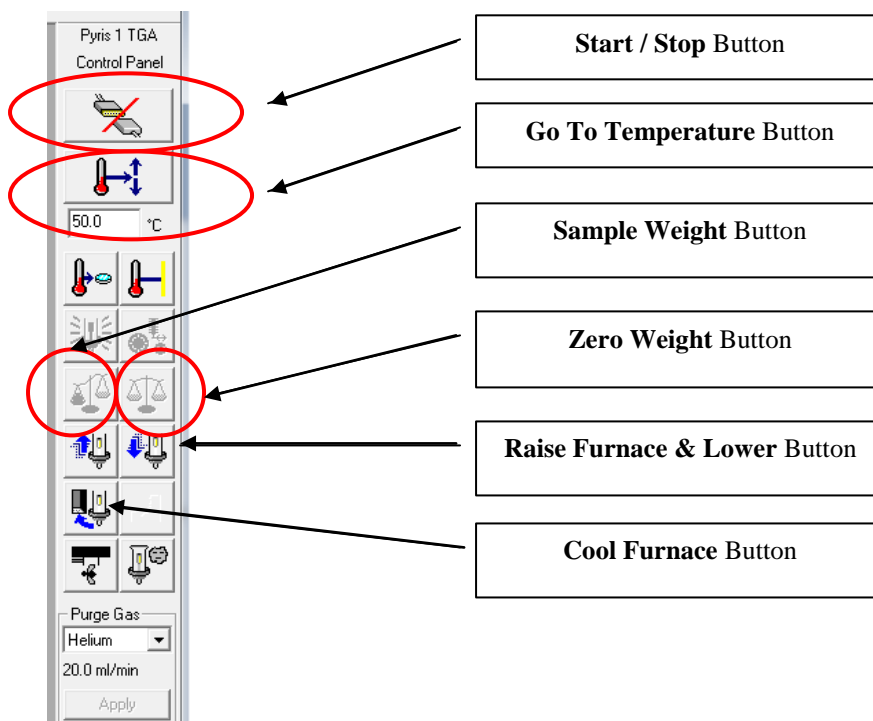
#### 2) Click Start/Program/Pyris/Pyris Manager to turn on Pyris software

- i) Wait for 20 seconds, click on the connection button on Pyris Manager bar to establish connection between Pyris 1 TGA system and computer.

ii)



- 3) Turn on sample purge gas, block purge gas and compressed air by turning regulator clockwise. (*Note: Typical purge gas- Nitrogen Gas - set at 25 psi, Balance purge gas- set at 50 psi, Compressed Air- set at 25 psi*).

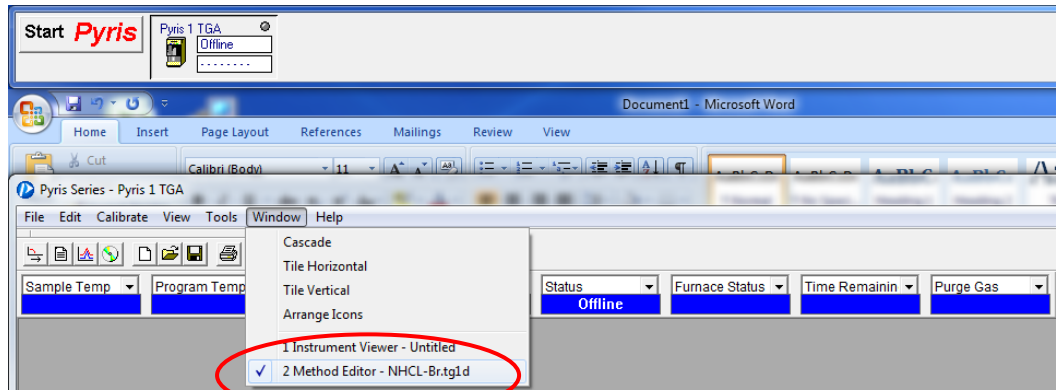


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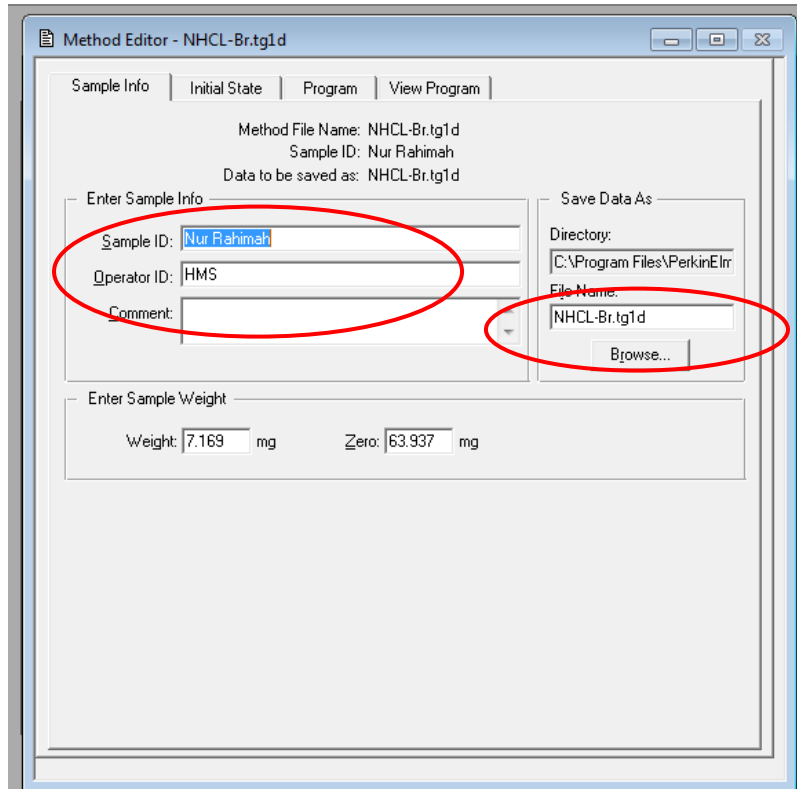
## Part 2 : Method Set Up Sample Run

*Method Editor* is used to set up method for sample run.

- 1) Click *Window/Method Editor* to start.




- 2) Click on *Sample Info* tab on the *Method Editor window*, input sample information accordingly into the related columns, such as *Sample ID*, *Operator ID* and *File Name*.




(Note 1: Browse button is used to specify alternative data saving path if default folder is not preferred).

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- 3) Allow Pyris 1 TGA system achieve equilibration and stability; gently hang an empty sample pan onto hangdown wire with the use of sample platform.

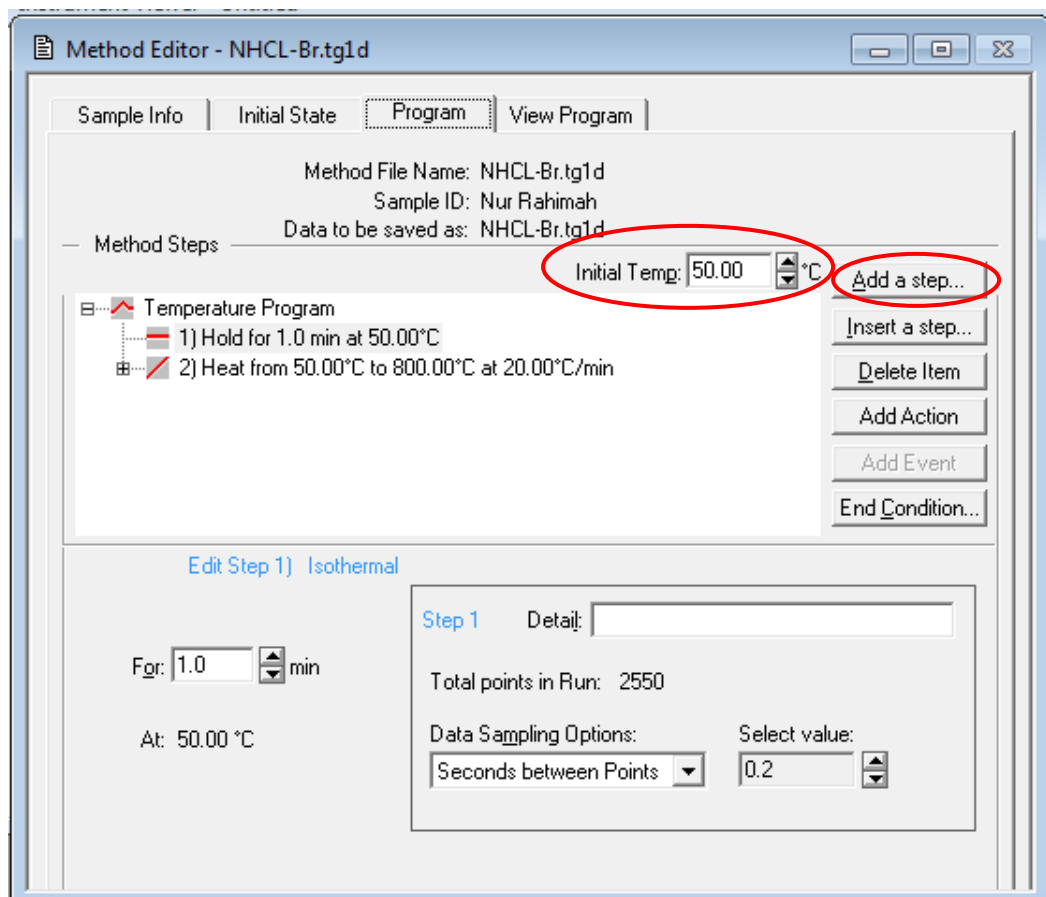
- 4) Monitor the weight reading, once it has stabilized, click **Zero Weight** button  on Control Panel tab to tare the weight.

- 5) Load sample into the empty sample pan, wait for the weight to stabilize and press **sample weight**

button  to read the weight of sample in milligram.

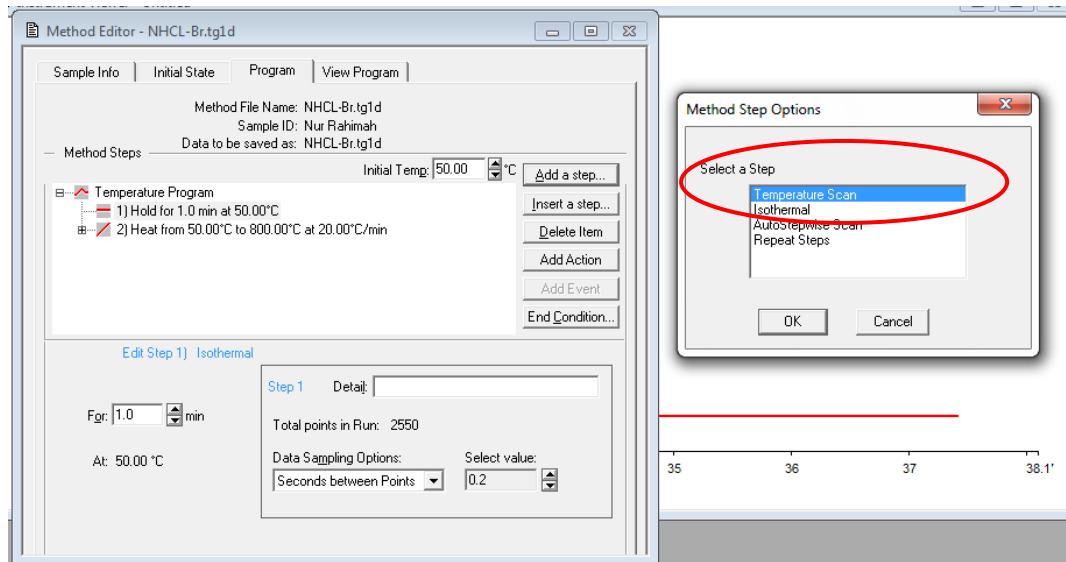
**WARNING:** Always load and unload sample and calibration materials into the TGA sample pan with the used of sample platform and hang the sample pan onto hangdown wire gently. Do not open the Balance Chamber. If weight changes become unstable, please contact our service representative.

- 6) Next, Click on **Program** tab to create method for sample analysis. Key in method initial temperature.



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- 7) Create new temperature program by clicking **Add a Step** button to create a new method step.

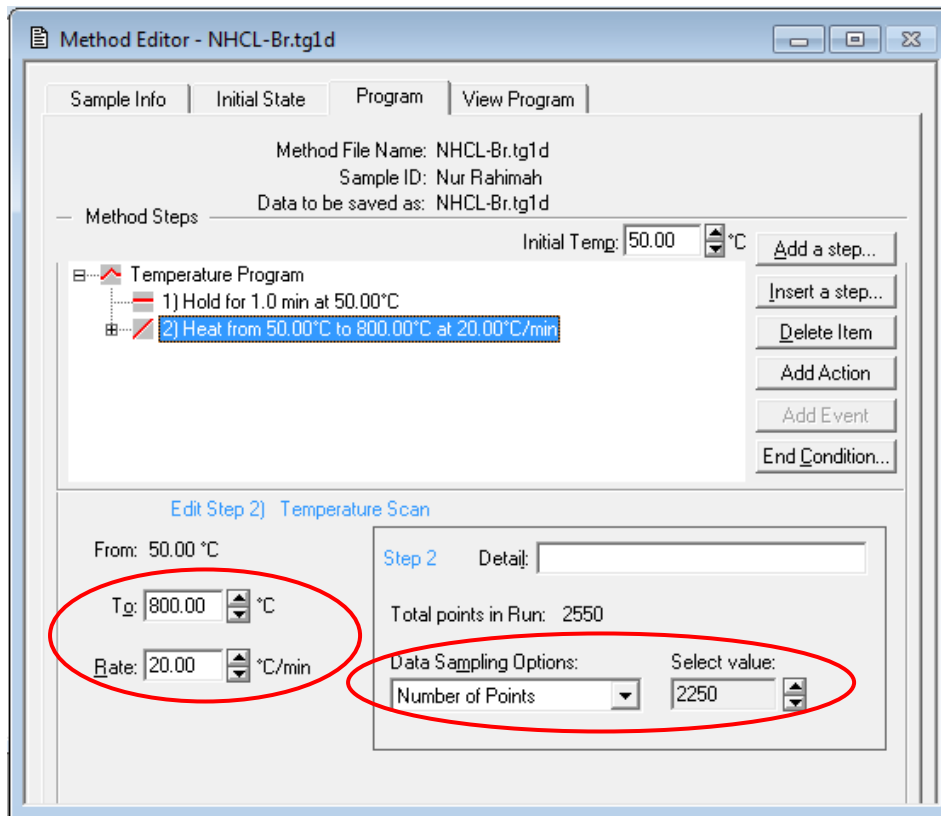


**Note :**

Temperature Scan	Heating and cooling temperature setting
Isothermal	Constant temperature setting
Repeat Steps	Duplicate steps in method program

- 8) For Temperature Scan Step, input final temperature, heating/cooling rate and data sampling options in respective columns.

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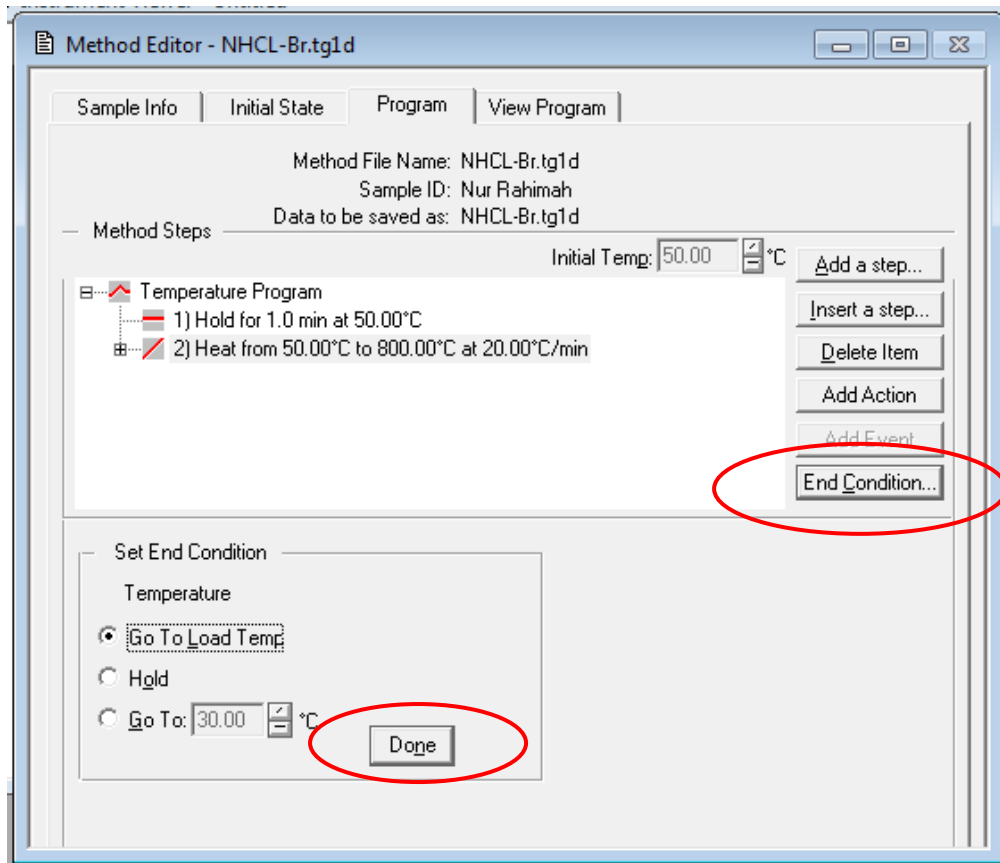
Note: There are two data sampling options :-

Number of Points	Total number of data points for the highlighted step per second. (One point taken at each second).
Second between Points	Normally set lower value, for test with faster reaction rate. (Example : 0.5 in order to take 2 data points per second).

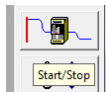
- 9) For Isothermal step, input **Initial temperature** and **Holding Time** in respective columns.
- 10) By default, at the end of the test (end of last temperature program step), the furnace will be brought back to default load temperature (<50°C) for safety and convenient sample loading purpose.


In order to change default end temperature, click the **End Condition** button and change the temperature setting accordingly.

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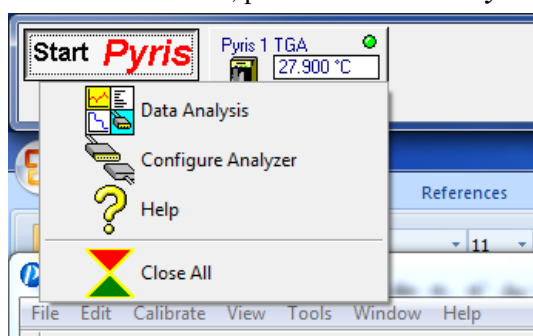


11) To save the newly created method, go to **File / Save Method As**.

12) After TGA system and balance achieve equilibrium, click  to start the analysis.

(**CAUTION** :  Loading and unloading of testing material should be carried out at room temperature or 50°C. The temperature of the furnace can reach as high as 1000°C. Always use tweezers to remove sample pan).

13) After the end of run, proceed to **Data Analysis** for calculation. Click **Start Pyris / Data Analysis**.



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- 14) Remove sample and reference pans after the end of analysis. If you wish to continue with second sample, just clean the sample pan, load the next sample and start another run.


**(REMEMBER : Always load and unload testing material at room temperature or 50°C)**

### **Part 3 : Proper way of Shutting Down STA System**


- 1) Bring the furnace temperature down to below 50°C, let the device to return to room temperature.
- 2) Turn off computer, DSC system and purge gas in sequence.

### **Part 4 : Maintenance**

Step 1	Furnace cleaning
Step 2	Sample pan cleaning

- 1) Click the ***Clean Furnace*** button  at the Control Panel. System will perform furnace cleaning by heating up to 900°C. As the furnace cleaning is completed, furnace will be moved to cool position and cool back to load temperature (30°C) with air cooling.

- 2) Purge the TGA with oxygen or compressed air and bring the furnace temperature to 900°C by

clicking the ***Go To Temp*** button  after key in 900°C in its entry box. Hold the furnace at 900°C for 5 to 10 minutes before cool it down to 30°C by clicking the ***Go To Temp*** button after key in 30°C in its entry box.

**(Note : Do not perform this without oxygen or compressed air purging).**

- 3) Always ensure balance purge (50 psi) is higher than sample purge (25 psi) to prevent sample back stream. **(Do not attempt to open the balance chamber!).**

## **4. PROSEDUR BERKAITAN**

Instrument Usage/Service Application Procedure

GP-I-001

## **5. RUJUKAN SEMAKAN**

SEMAKAN	DISEDIAKAN	DISEMAK	TARIKH	CATATAN
1	Hashim Bin Mohammad Salleh.	Muhammad Fauzan Bin Zainudin	09.08.2018	Isu kali pertama