



**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

**1.0 OBJECTIVE:**

To lay down the procedure for Operation & Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space).

**2.0 SCOPE:**

This SOP is applicable to operation and calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) in the quality control department.

**3.0 RESPONSIBILITY:**

Officer, Executive, Sr. Executive- Quality control Department.  
Head- Quality Control Department.

**4.0 PROCEDURE:**

**4.1 Operation:**

**Preliminary Check:**

- 4.1.1 Ensure that the instrument is clean & free from dust.
- 4.1.2 Ensure that incoming air & gas pressure is more than 5.0 Kg/ cm<sup>2</sup>.
- 4.1.3 Switch ON the nitrogen, hydrogen and air to check the supply.
- 4.1.4 Ensure that the gas pressures are as below on the gas panel or as per respective standard test procedure;
  - Carrier gas (Nitrogen/Helium): 30.0 mL/min
  - Zero air: 400.0 mL/min
  - Hydrogen: 40.0 mL/min
- 4.1.5 Place the column nuts, graphite ferrules on the ends of the desired capillary column. In case of new column, cut 1 mm at each end by using column cutter.
- 4.1.6 Connect the capillary column to the injection port (push 30.0 mm to injection port) and the detector port (push the column 70.0 mm to FID). Tight the column nut by hand first, then 2 turns with a wrench (spanner).
- 4.1.7 Attach the column to the column holder.



**STANDARD OPERATING PROCEDURE**

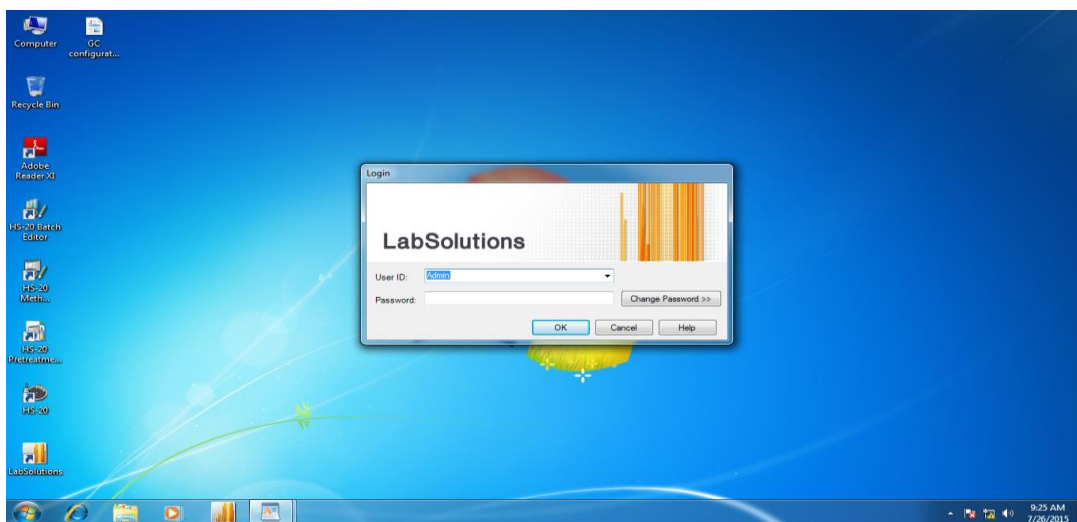
|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

- 4.1.8 Check and ensure that there is no leakage in all joints.
- 4.1.9 Check and ensure that the syringe is fixed in proper position as required.
- 4.1.10 Check and ensure that the gas supply is available as required for the system operation.
- 4.1.11 Switch ON the GC & CBM (Communication Bus) and wait for inter communication of system, display on GC will show link ok with a beep.
- 4.1.12 Turn on the personal computer attached to GC The system setup windows automatically. The windows Program Manager will appear on the screen.
- 4.1.13 **Startup procedure of LAB SOLUTION Software:**
- 4.1.13.1 Main window of PC shows icons in the left side of monitor.



**LabSolutions.Ink**

- 4.1.13.2 Double Click on LAB SOLUTION icon. Enter the user name and Password then ok.





# PHARMA DEVILS

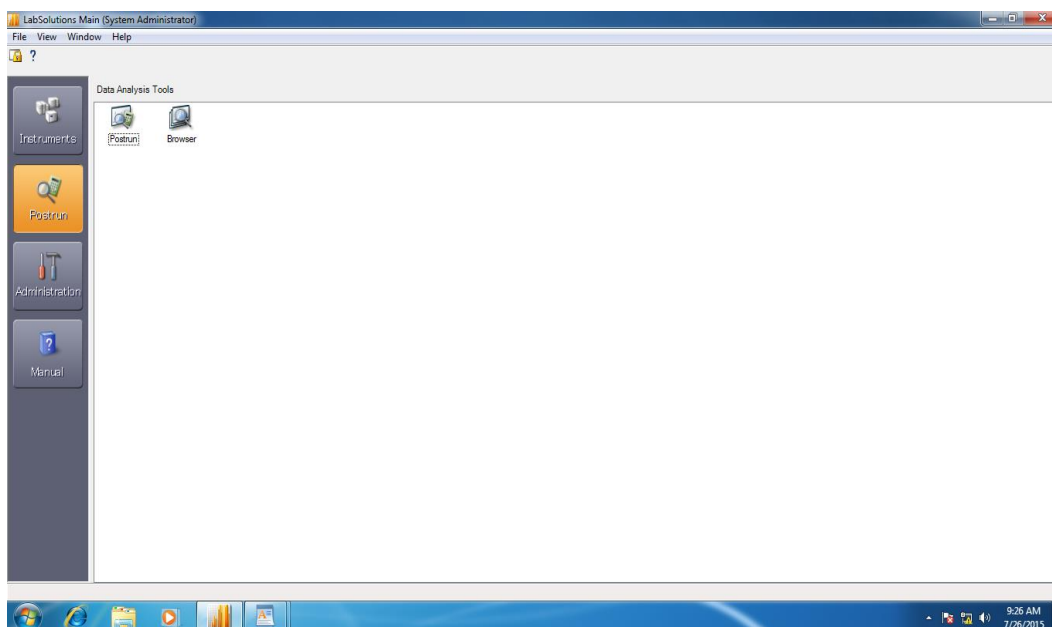
QUALITY CONTROL DEPARTMENT

## STANDARD OPERATING PROCEDURE

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
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| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

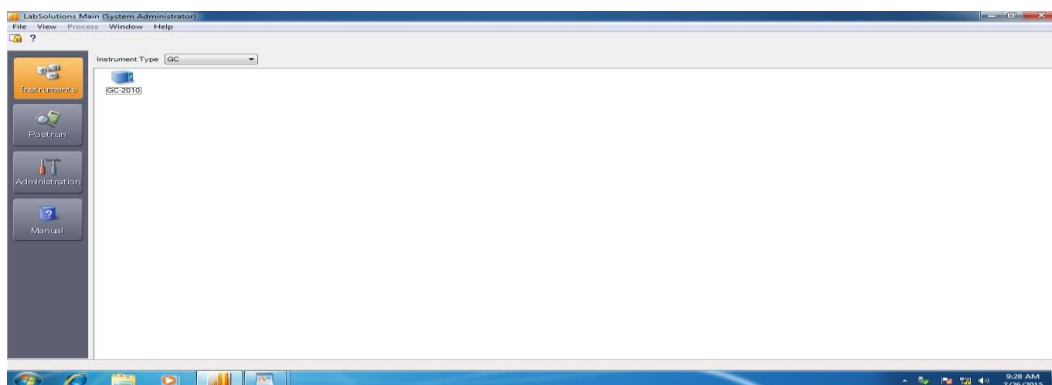
4.1.13.3 Shall display on the screen.

4.1.13.4



4.1.13.5

Select the instrument and then double click on the instrumentation.





# PHARMA DEVILS

## QUALITY CONTROL DEPARTMENT

### STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

**Effective Date:**

**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

4.1.13.6

| Item               | Value | Ctrl   |
|--------------------|-------|--------|
| SPL2 Temperature   | 34.0  |        |
| SPL2 Pressure      | 0.0   |        |
| Total Flow         | 0.2   |        |
| Purge Flow         | 0.0   | On Off |
| Primary Pressure   | 307   |        |
| Column Temperature | 24.0  |        |
| FID1 Temperature   | 25.2  |        |
| FID1 Makeup Flow   | 0.0   | On Off |
| FID1 H2 Flow       | 0.0   | On Off |
| FID1 Air Flow      | 0.0   | On Off |
| APC1 Pressure      | 0.0   | On Off |
| APC2 Pressure      | 0.0   | On Off |
| APC3 Pressure      | 0.0   | On Off |

4.1.13.7

Enter the Oven temperature, Detector Temperature, and select the injector Mode Split & Split less as per requirement.

4.1.13.8

After achieving of set FID Detector temperature click on "Ignite" to flame ignition.

4.1.13.9

Select system configuration and click to set for configured modules setting with system.

4.1.13.10

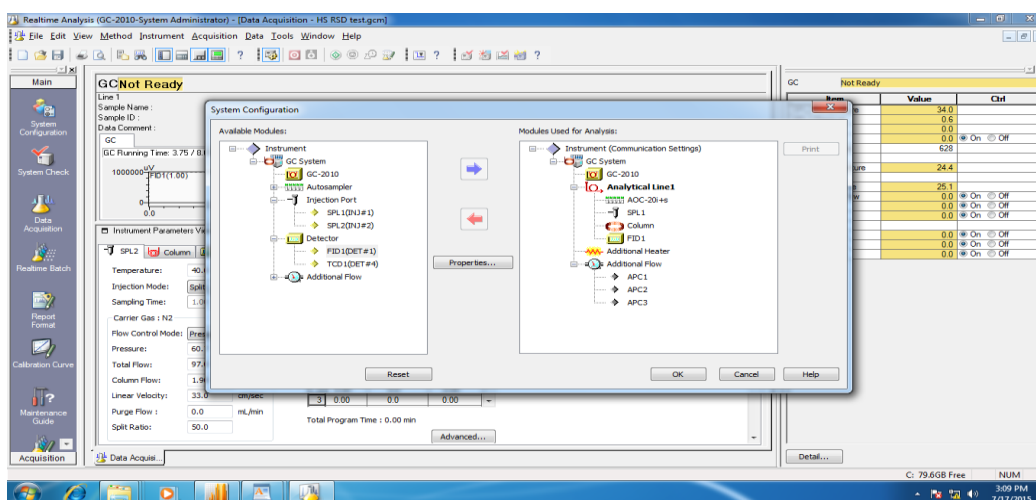
| Item               | Value | Ctrl   |
|--------------------|-------|--------|
| SPL2 Temperature   | 34.0  |        |
| SPL2 Pressure      | 0.0   |        |
| Total Flow         | 0.1   |        |
| Purge Flow         | 0.0   | On Off |
| Primary Pressure   | 166   |        |
| Column Temperature | 24.0  |        |
| FID1 Temperature   | 25.2  |        |
| FID1 Makeup Flow   | 0.0   | On Off |
| FID1 H2 Flow       | 0.0   | On Off |
| FID1 Air Flow      | 0.0   | On Off |
| APC1 Pressure      | 0.0   | On Off |
| APC2 Pressure      | 0.0   | On Off |
| APC3 Pressure      | 0.0   | On Off |



### STANDARD OPERATING PROCEDURE

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|------------------------------------------------------------------------------------------------------------|------------------------|
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| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

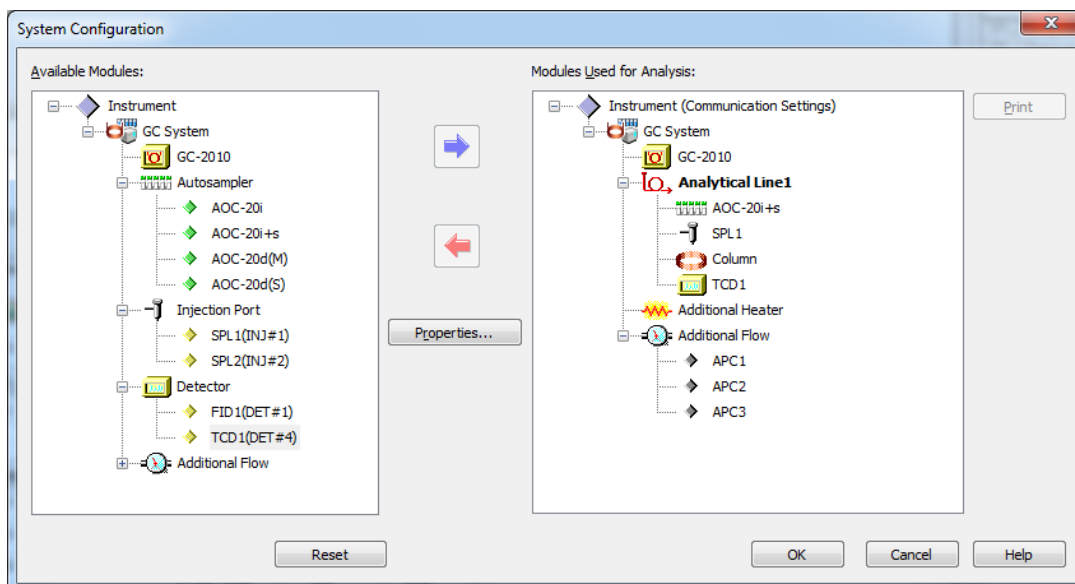
4.1.13.11



4.1.13.12

This window Configuration only Liquid Sample AOC 20i+s, SPL1 (liquid inject port) and FID.

4.1.13.13



4.1.13.14

This window Configuration is only for Liquid Sample AOC 20i+s, SPL1 (liquid inject port) and TCD.



**STANDARD OPERATING PROCEDURE**

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**Effective Date:**

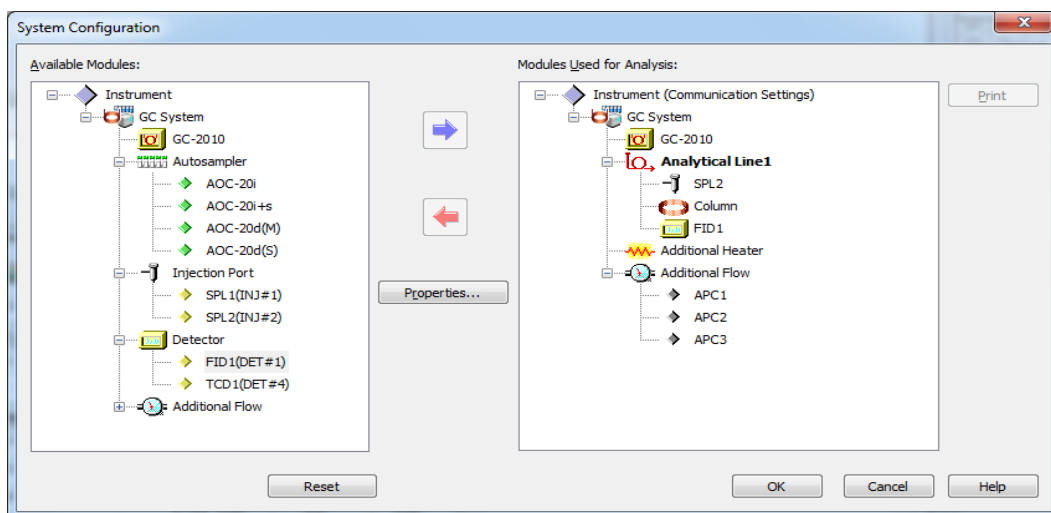
**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

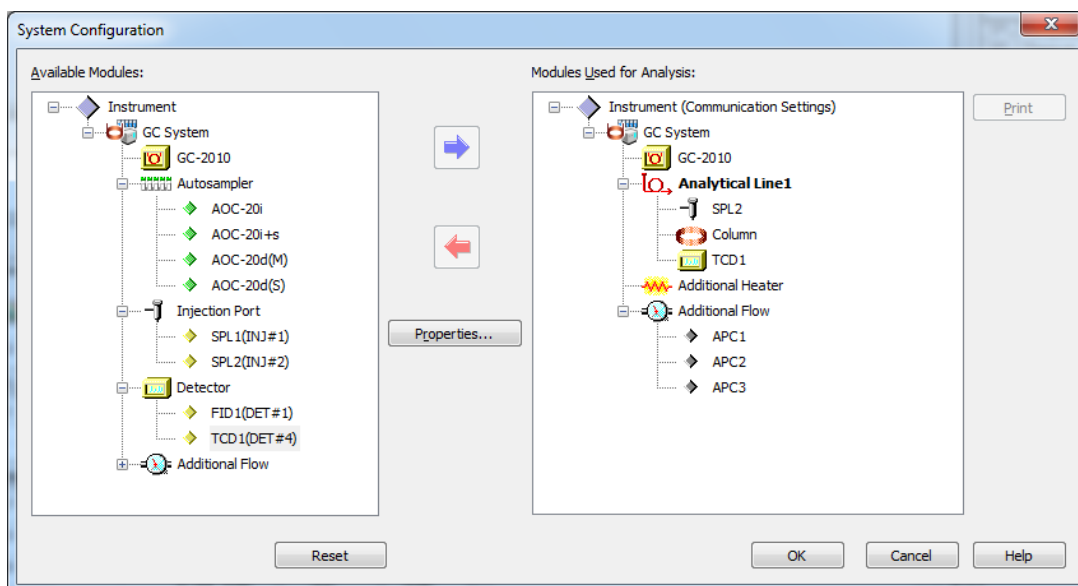
4.1.13.15



4.1.13.16

This window Configuration SPL2 (HS-20), Column oven and FID.

4.1.13.17



4.1.13.18

This window Configuration SPL2 (HS-20), Col oven and TCD.

4.1.13.19

As per required configuration then OK.

**4.1.14**

**Creating the Method (Through Software).**

4.1.14.1

Click on file then select the new method.



# PHARMA DEVILS

## QUALITY CONTROL DEPARTMENT

### STANDARD OPERATING PROCEDURE

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**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

**Effective Date:**

**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

4.1.14.2

| Item             | Value | Setting | Units  | Ctrl                                                     |
|------------------|-------|---------|--------|----------------------------------------------------------|
| SPL2 Temperature | 34.0  | 30.0    | C      |                                                          |
| SPL2 Pressure    | 120.3 | 120.3   | kPa    |                                                          |
| Total Flow       | 178.1 | 178.2   | mL/min |                                                          |
| Purge Flow       | 3.0   | 3.0     | mL/min | <input type="checkbox"/> On <input type="checkbox"/> Off |
| Primary Pressure | 598   |         | kPa    |                                                          |
| Column Temporal  | 60.2  | 60.0    | C      |                                                          |
| FID1 Temperature | 133.4 | 260.0   | C      |                                                          |
| FID1 Makeup Fl   | 30.0  | 30.0    | mL/min | <input type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 H2 Flow     | 0.1   | 40.0    | mL/min | <input type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 Air Flow    | 0.0   | 400.0   | mL/min | <input type="checkbox"/> On <input type="checkbox"/> Off |
| APC1 Pressure    | 29.9  | 30.0    | kPa    | <input type="checkbox"/> On <input type="checkbox"/> Off |
| APC2 Pressure    | 0.0   | 0.0     | kPa    | <input type="checkbox"/> On <input type="checkbox"/> Off |
| APC3 Pressure    | 0.0   | 0.0     | kPa    | <input type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 Detector    |       |         |        | <input type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 Flame       |       |         |        | <input type="checkbox"/> On <input type="checkbox"/> Off |
| Carrier Gas      |       |         |        | <input type="checkbox"/> On <input type="checkbox"/> Off |

4.1.14.3

| Item             | Value | Setting | Units  | Ctrl                                                     |
|------------------|-------|---------|--------|----------------------------------------------------------|
| SPL2 Temperature | 34.0  | 30.0    | C      |                                                          |
| SPL2 Pressure    | 120.3 | 120.3   | kPa    |                                                          |
| Total Flow       | 178.2 | 178.2   | mL/min |                                                          |
| Purge Flow       | 3.0   | 3.0     | mL/min | <input type="checkbox"/> On <input type="checkbox"/> Off |
| Primary Pressure | 591   |         | kPa    |                                                          |
| Column Temporal  | 60.0  | 60.0    | C      |                                                          |
| FID1 Temperature | 269.7 | 260.0   | C      |                                                          |
| FID1 Makeup Fl   | 30.0  | 30.0    | mL/min | <input type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 H2 Flow     | 40.0  | 40.0    | mL/min | <input type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 Air Flow    | 399.7 | 400.0   | mL/min | <input type="checkbox"/> On <input type="checkbox"/> Off |
| APC1 Pressure    | 30.0  | 30.0    | kPa    | <input type="checkbox"/> On <input type="checkbox"/> Off |
| APC2 Pressure    | 0.0   | 0.0     | kPa    | <input type="checkbox"/> On <input type="checkbox"/> Off |
| APC3 Pressure    | 0.0   | 0.0     | kPa    | <input type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 Detector    |       |         |        | <input type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 Flame       |       |         |        | <input type="checkbox"/> On <input type="checkbox"/> Off |
| Carrier Gas      |       |         |        | <input type="checkbox"/> On <input type="checkbox"/> Off |

4.1.14.4

Click the New Method then OK.



# PHARMA DEVILS

## QUALITY CONTROL DEPARTMENT

### STANDARD OPERATING PROCEDURE

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**Effective Date:**

**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

#### 4.1.14.5

| Item               | Value | Setting | Units  | Ctrl   |
|--------------------|-------|---------|--------|--------|
| SPL2 Temperature   | 34.0  | 30.0    | C      |        |
| SPL2 Pressure      | 120.3 | 120.3   | kPa    |        |
| Total Flow         | 178.2 | 178.2   | mL/min |        |
| Purge Flow         | 3.0   | 3.0     | mL/min | On Off |
| Primary Pressure   | 585   |         | kPa    |        |
| Column Temperature | 60.0  | 60.0    | C      |        |
| FID1 Temperature   | 259.8 | 260.0   | C      |        |
| FID1 Makeup Flo    | 30.0  | 30.0    | mL/min | On Off |
| FID1 H2 Flow       | 40.0  | 40.0    | mL/min | On Off |
| FID1 Ar Flow       | 399.7 | 400.0   | mL/min | On Off |
| APC1 Pressure      | 30.0  | 30.0    | kPa    | On Off |
| APC2 Pressure      | 0.0   | 0.0     | kPa    | On Off |
| APC3 Pressure      | 0.0   | 0.0     | kPa    | On Off |
| FID1 Detector      |       |         |        | On Off |
| FID1 Flame         |       |         |        | On Off |
| Carrier Gas        |       |         |        | On Off |

#### 4.1.14.6

| Item               | Value | Setting | Units  | Ctrl   |
|--------------------|-------|---------|--------|--------|
| SPL2 Temperature   | 34.0  | 30.0    | C      |        |
| SPL2 Pressure      | 120.3 | 120.3   | kPa    |        |
| Total Flow         | 178.2 | 178.2   | mL/min |        |
| Purge Flow         | 3.0   | 3.0     | mL/min | On Off |
| Primary Pressure   | 576   |         | kPa    |        |
| Column Temperature | 60.0  | 60.0    | C      |        |
| FID1 Temperature   | 260.0 | 260.0   | C      |        |
| FID1 Makeup Flo    | 30.0  | 30.0    | mL/min | On Off |
| FID1 H2 Flow       | 40.0  | 40.0    | mL/min | On Off |
| FID1 Ar Flow       | 399.8 | 400.0   | mL/min | On Off |
| APC1 Pressure      | 30.0  | 30.0    | kPa    | On Off |
| APC2 Pressure      | 0.0   | 0.0     | kPa    | On Off |
| APC3 Pressure      | 0.0   | 0.0     | kPa    | On Off |
| FID1 Detector      |       |         |        | On Off |
| FID1 Flame         |       |         |        | On Off |
| Carrier Gas        |       |         |        | On Off |

#### 4.1.14.7

### INJECTION:

- ❖ Temperature (as specified in the method)
- ❖ Injection mode (Split)
- ❖ Sampling time (as required)
- ❖ Flow control mode (Pressure)
- ❖ Pressure (as required)





# PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

## STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

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**Effective Date:**

**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

- ❖ Total flow/Column flow (as specified in the method)
- ❖ Linear velocity
- ❖ Purge flow
- ❖ Split ratio (as specified in method)
- ❖ Click to Set (for column selection as required)
- ❖ Injection program (Rate, Pressure, Hold time)

4.1.14.8

The screenshot displays the Shimadzu GC-2010 Plus software interface. The main window is titled "GC Not Ready" and shows various instrument parameters and a data table.

**Instrument Parameters View:**

- Temperature: 50.0 C
- Injection Mode: Split
- Sampling Time: 1.00 min
- Carrier Gas: N2
- Flow Control Mode: Pressure
- Pressure: 100.0 kPa
- Total Flow: 50.0 mL/min
- Column Flow: 4.15 mL/min
- Linear Velocity: 56.8 cm/sec

**INI Programs:**

| Rate | Pressure | Hold Time |
|------|----------|-----------|
| 0    | 100.0    | 0.00      |
| 1    | 0.0      | 0.00      |
| 2    | 0.0      | 0.00      |
| 3    | 0.0      | 0.00      |

**GC Parameters Table:**

| Item              | Value | Setting | Units  | Ctrl   |
|-------------------|-------|---------|--------|--------|
| SPL2 Temperatur   | 50.0  | 50.0    | C      |        |
| SPL2 Pressure     | 100.0 | 100.0   | kPa    |        |
| Total Flow        | 178.2 | 178.2   | mL/min |        |
| Purge Flow        | 3.0   | 3.0     | mL/min | On Off |
| Primary Pressure  | 589   |         | kPa    |        |
| Column Temperatur | 60.0  | 60.0    | C      |        |
| FID1 Temperatur   | 259.0 | 260.0   | C      |        |
| FID1 Makeup Flo   | 30.0  | 30.0    | mL/min | On Off |
| FID1 H2 Flow      | 40.0  | 40.0    | mL/min | On Off |
| FID1 Ar Flow      | 399.7 | 400.0   | mL/min | On Off |
| APC1 Pressure     | 30.0  | 30.0    | kPa    | On Off |
| APC2 Pressure     | 0.0   | 0.0     | kPa    | On Off |
| APC3 Pressure     | 0.0   | 0.0     | kPa    | On Off |
| FID1 Detector     |       |         |        | On Off |
| FID1 Flame        |       |         |        | On Off |
| Carrier Gas       |       |         |        | On Off |



# PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

## STANDARD OPERATING PROCEDURE

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

### 4.1.14.9 COLUMN:

- ❖ Temperature (as specified in the method)
- ❖ Equilibrium time (1.0 min)
- ❖ Enter the Column Length, Film thickness, Inner diameter & Column Maximum Temperature.

The screenshot displays the Shimadzu GC-2010 Plus software interface. The main window is titled "GCReady" and shows the following parameters:

- Line 1
- Sample Name:
- Sample ID:
- Data Comment:
- GC
- GC Running Time: 11.82 / 60.00 min FID1: -36161uV
- Max Intensity: 973.061
- Instrument Parameters View: Normal, Advanced, End Time: 60.00 min
- Temperature: 60.0 C
- Equilibration Time: 3.0 min
- Column Information (BP-624)
- Column ID:
- Installation Date: 15/07/13
- Column Max Temp.: 230 C
- Length: 30.0 m
- Inner Diameter: 0.32 mm ID
- Film Thickness: 1.80 um
- Column Oven Temperature Program table:

| Rate | Temperature | Hold Time |
|------|-------------|-----------|
| 0    | 25.0        | 0.00      |
| 1    | 0.00        | 0.00      |
| 2    | 0.00        | 0.00      |
| 3    | 0.00        | 0.00      |

The right-hand panel shows a table of instrument parameters:

| Item              | Value | Setting | Units  | Ctrl   |
|-------------------|-------|---------|--------|--------|
| SPL2 Temperature  | 34.0  | 30.0    | C      |        |
| SPL2 Pressure     | 120.3 | 120.3   | kPa    |        |
| Total Flow        | 178.2 | 178.2   | mL/min |        |
| Purge Flow        | 3.0   | 3.0     | mL/min | On Off |
| Primary Pressure  | 573   |         | kPa    |        |
| Column Temporal   | 60.0  | 60.0    | C      |        |
| FID1 Temperature  | 260.0 | 260.0   | C      |        |
| FID1 Make-up Flow | 30.0  | 30.0    | mL/min | On Off |
| FID1 H2 Flow      | 40.0  | 40.0    | mL/min | On Off |
| FID1 Air Flow     | 399.8 | 400.0   | mL/min | On Off |
| APC1 Pressure     | 30.0  | 30.0    | kPa    | On Off |
| APC2 Pressure     | 0.0   | 0.0     | kPa    | On Off |
| APC3 Pressure     | 0.0   | 0.0     | kPa    | On Off |
| FID1 Detector     |       |         |        | On Off |
| FID1 Flame        |       |         |        | On Off |
| Carrier Gas       |       |         |        | On Off |



# PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

## STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

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**Effective Date:**

**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

### 4.1.14.10 DETECTOR:

- ❖ Temperature
- ❖ Sampling ratio (100)
- ❖ Stop Time
- ❖ Programming.

The screenshot displays the 'Realtime Analysis (GC-2010-System Administrator) - [Data Acquisition - Untitled]' window. The main panel shows 'GCReady' status with a chromatogram plot. The 'Instrument Parameters View' is active, showing settings for Temperature (25.0 C), Sampling Rate (40 msec), Stop Time (60.00 min), and Delay Time (0.00 min). A 'Flow Program' table is visible below the parameters:

| Rate | Flow | Hold Time |
|------|------|-----------|
| 0    | 30.0 | 0.00      |
| 1    | 0.00 | 0.00      |
| 2    | 0.00 | 0.00      |
| 3    | 0.00 | 0.00      |

On the right, a 'GC Ready' table lists various parameters and their settings:

| Item             | Value | Setting | Units  | Ctrl                                                                |
|------------------|-------|---------|--------|---------------------------------------------------------------------|
| SPL2 Temperature | 34.0  | 30.0    | C      |                                                                     |
| SPL2 Pressure    | 120.3 | 120.3   | kPa    |                                                                     |
| Total Flow       | 178.2 | 178.2   | mL/min |                                                                     |
| Purge Flow       | 3.0   | 3.0     | mL/min | <input checked="" type="checkbox"/> On <input type="checkbox"/> Off |
| Primary Pressure | 553   |         | kPa    |                                                                     |
| Column Temperat  | 60.0  | 60.0    | C      |                                                                     |
| FID1 Temperature | 260.0 | 260.0   | C      |                                                                     |
| FID1 Makeup Flo  | 30.0  | 30.0    | mL/min | <input checked="" type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 H2 Flow     | 40.0  | 40.0    | mL/min | <input checked="" type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 Ar Flow     | 399.8 | 400.0   | mL/min | <input checked="" type="checkbox"/> On <input type="checkbox"/> Off |
| APC1 Pressure    | 30.0  | 30.0    | kPa    | <input checked="" type="checkbox"/> On <input type="checkbox"/> Off |
| APC2 Pressure    | 0.0   | 0.0     | kPa    | <input checked="" type="checkbox"/> On <input type="checkbox"/> Off |
| APC3 Pressure    | 0.0   | 0.0     | kPa    | <input checked="" type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 Detector    |       |         |        | <input checked="" type="checkbox"/> On <input type="checkbox"/> Off |
| FID1 Flame       |       |         |        | <input checked="" type="checkbox"/> On <input type="checkbox"/> Off |
| Carrier Gas      |       |         |        | <input checked="" type="checkbox"/> On <input type="checkbox"/> Off |

The bottom status bar shows 'C: 79.2GB Free NUM SCRL 10:58 AM 7/26/2015'.



# PHARMA DEVILS

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**Effective Date:**

**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

### 4.1.14.11 GENERAL :-

- ❖ Ready Check
- ❖ Heat Unit
- ❖ Injection Flow
- ❖ FID.

| Item               | Value | Setting | Units  | Ctrl   |
|--------------------|-------|---------|--------|--------|
| SPL2 Temperature   | 34.0  | 30.0    | C      |        |
| SPL2 Pressure      | 120.3 | 120.3   | kPa    |        |
| Total Flow         | 178.2 | 178.2   | mL/min |        |
| Purge Flow         | 3.0   | 3.0     | mL/min | On Off |
| Primary Pressure   | 553   |         | kPa    |        |
| Column Temperature | 60.0  | 60.0    | C      |        |
| FID1 Temperature   | 260.0 | 260.0   | C      |        |
| FID1 Makeup Flow   | 30.0  | 30.0    | mL/min | On Off |
| FID1 H2 Flow       | 40.0  | 40.0    | mL/min | On Off |
| FID1 Air Flow      | 399.8 | 400.0   | mL/min | On Off |
| APC1 Pressure      | 30.0  | 30.0    | kPa    | On Off |
| APC2 Pressure      | 0.0   | 0.0     | kPa    | On Off |
| APC3 Pressure      | 0.0   | 0.0     | kPa    | On Off |
| FID1 Detector      |       |         |        | On Off |
| FID1 Range         |       |         |        | On Off |
| Carrier Gas        |       |         |        | On Off |

4.1.14.12 After setting the above parameter, click on File and save the method file as per the required name.

4.1.14.13 To carry out the analysis open the method file and check the parameters as per method requirement.

4.1.14.14 Click on View to monitor Instrument parameters; the window will be displayed on the right side of the screen.

4.1.14.15 Click down load icon and wait for parameter setting.

4.1.14.16 Click on the System ON icon menu.

4.1.14.17 System will start automatically. Display will show method parameter on right side of window in two colour (yellow & black). The status of the system will be displayed as **System not ready**.



## STANDARD OPERATING PROCEDURE

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

4.1.14.18 When system becomes ready, yellow colour disappears and the window shows status as **Ready mode**.

4.1.14.19 Click flame ON to ignite the flame. It gives a minor sound when flame ignites. Similarly click ON to start the detector. Check the status of flame and detector. It should show ON status.

### 4.1.14.20 Startup Analysis:

4.1.14.20.1 Click to the single injection or Batch analysis icon feed the information as per requirement and check baseline correction.

#### 4.1.14.20.2 For Single Start

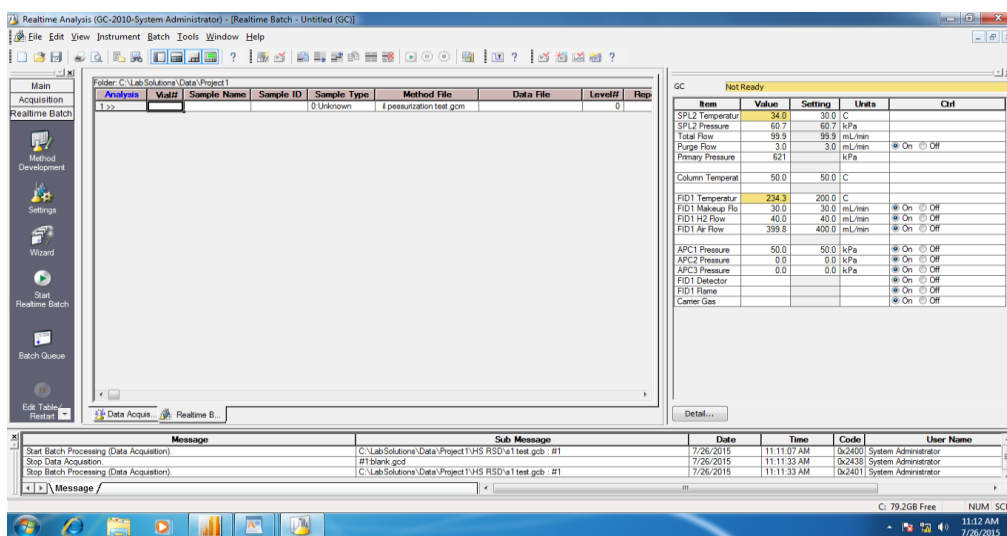
4.1.14.20.3 Click on the Single Run and input Sample Name, Sampled ID, Vial no, File Name, and Description in the sample login and then click OK.

4.1.14.20.4 Click zero GC to make base line correction.

4.1.14.20.5 Click to Start the single injection for run.

### 4.1.14.21 Creating the New Sequence.

4.1.14.21.1 Double Click the Real time Batch, File will be displayed on the screen. Select new batch file.



4.1.14.21.2 Click on file and open new batch file. Input Vial No., Sample name, Sample ID, Method file, Data file and Data description and then save batch file as per the required name.



# PHARMA DEVILS

## QUALITY CONTROL DEPARTMENT

### STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

**Effective Date:**

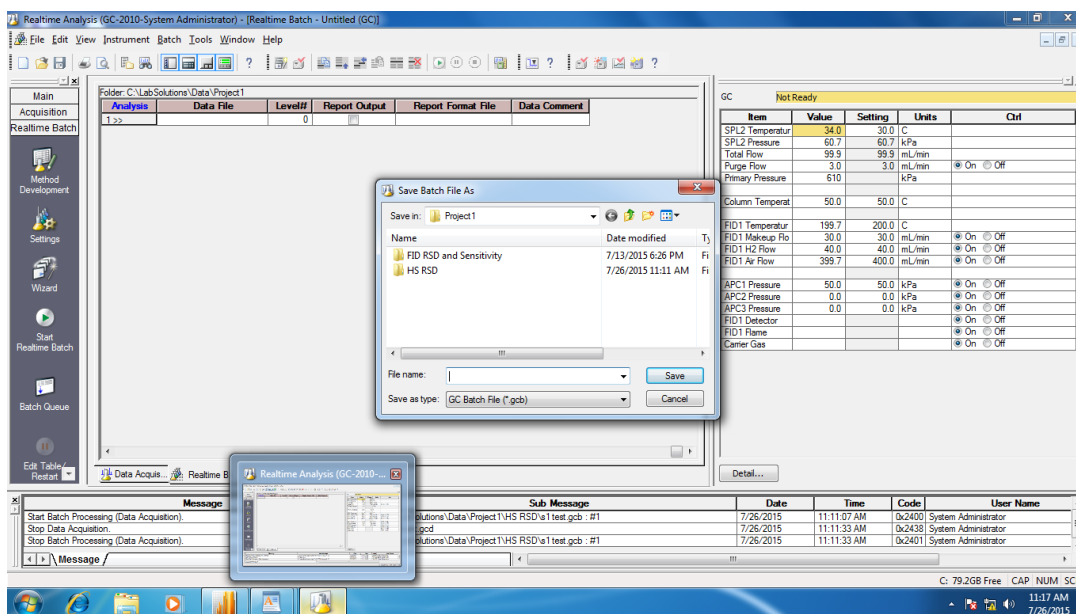
**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

#### 4.1.14.21.3

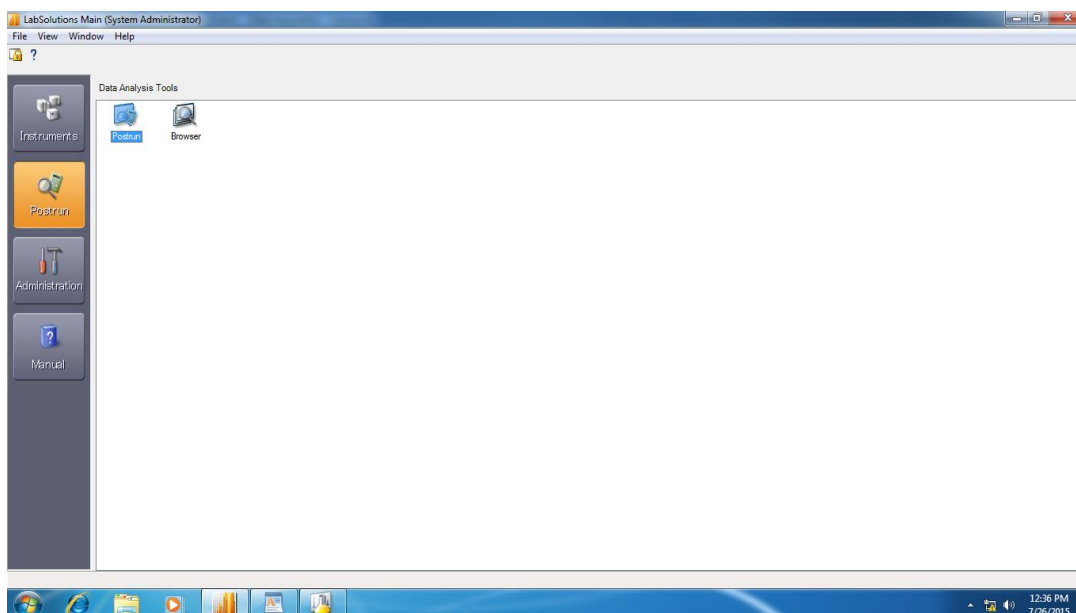


4.1.14.21.4 Click on File and save the sequence file as per the required name.

4.1.14.21.5 Click to start the batch-processing run.

#### 4.1.14.22 DATA PROCESSING:

4.1.14.22.1 After completion of the injections, click **GC Post run analysis** icon on the main window to open the data file.





# PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

## STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

**Effective Date:**

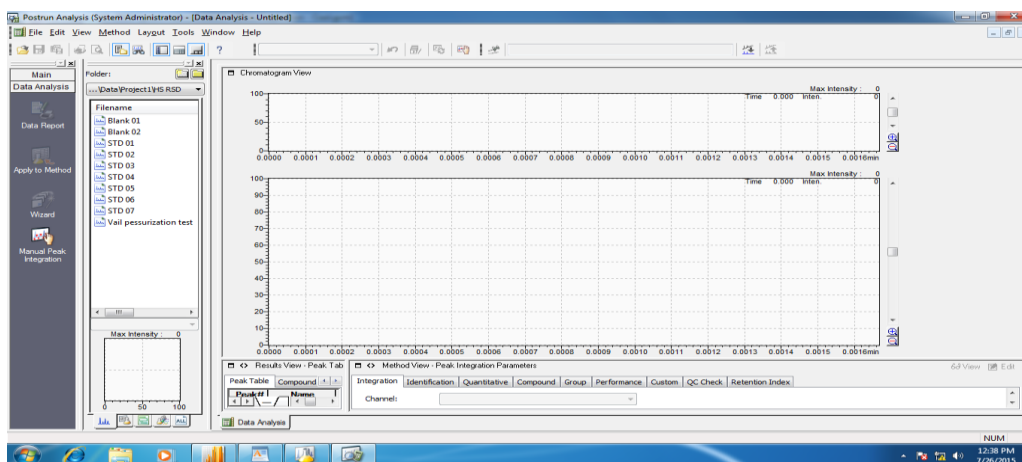
**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

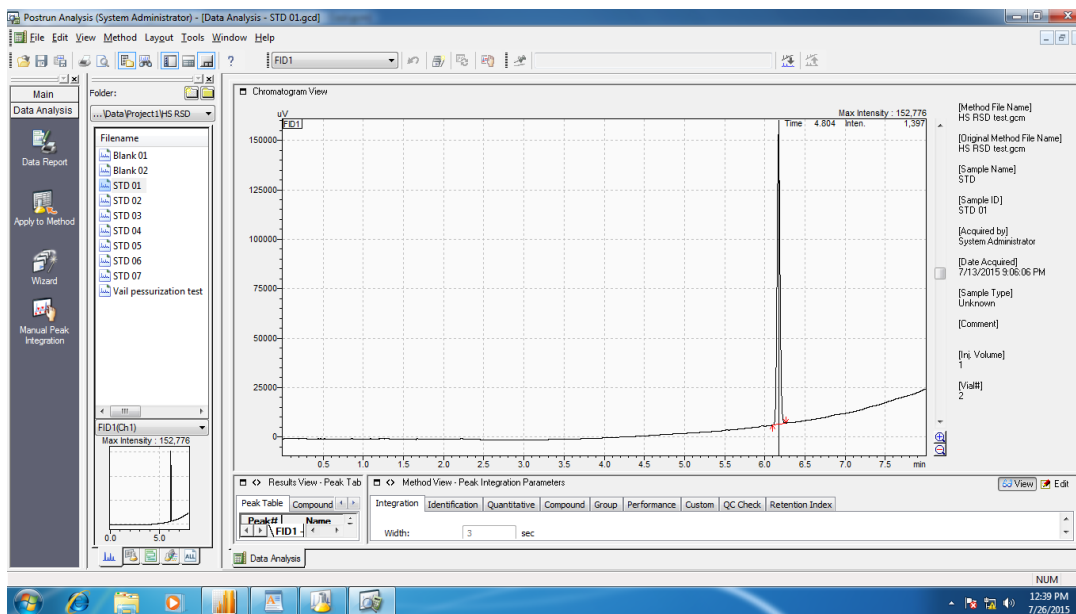
4.1.14.22.2



4.1.14.22.3

Select the File Double Click.

4.1.14.22.4



4.1.14.22.5

Then click Wizard.



# PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

## STANDARD OPERATING PROCEDURE

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

4.1.14.22.6

The screenshot shows the Postrun Analysis software interface. A 'Compound Table Wizard 1/5' dialog box is open, overlaid on a chromatogram. The wizard contains the following fields and options:

- Width: 3 sec
- Slope: 10000 u/min
- Drift: 0 u/min
- T. DBL: 1000 min
- Min. Area/Height: 100000 counts
- Calculated by:  Area  Height
- Buttons: < Back, Next >, Cancel, Help, Noise/Drift Calculation..., Advanced...

The background chromatogram shows a single sharp peak at approximately 3.562 minutes. The y-axis is labeled 'uV' and ranges from 0 to 150,000. The x-axis is labeled 'min' and ranges from 0.0 to 7.5. The peak is labeled 'FID1' and has a 'Max Intensity: 152,776'.

4.1.14.22.7 Click Program.

The screenshot shows the Postrun Analysis software interface with the 'Integration Time Program' dialog box open. The dialog box contains the following fields and options:

- Time (min): 0.000
- Command: [Empty]
- Value: 0
- Buttons: OK, Cancel, Load Data..., Simulate, Help

The background shows the same chromatogram as in the previous screenshot, with the peak at 3.562 minutes. The 'Integration Time Program' dialog box is positioned over the peak, and the 'Integration' tab is selected in the software's interface.





# PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

## STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

**Effective Date:**

**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

### 4.1.14.22.8 Enter integration parameters. And then Ok.

Postrun Analysis (System Administrator) - [Data Analysis - STD 01.gcd]

Chromatogram View

Max Intensity: 152,776

Time: 0.000 Inten: -893

Compound Table Wizard 2/5

| Select                              | Ret. Time | Area   | Height |
|-------------------------------------|-----------|--------|--------|
| <input checked="" type="checkbox"/> | 6.170     | 375769 | 144485 |

Select peaks you want to define by checking boxes on the peak table.

Max Intensity: 152,776

Time: 6.170 min

Peak Integration Parameters

Quantitative | Compound | Group | Performance | Custom | QC Check | Retention Index

NUM 12:42 PM 7/26/2015



# PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

## STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

**Effective Date:**

**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

### 4.1.14.22.9 Tick the Intergrated Peak. Then Next.

| Select                              | Ret. Time | Area   | Height |
|-------------------------------------|-----------|--------|--------|
| <input checked="" type="checkbox"/> | 6.170     | 375769 | 144485 |

Identification

Window/Band:  Window  Band

Window: 10 %

Default bandwidth: 0.01 min

Identification Method: Absolute RI

Peak Selection: All Peaks

Display not identified peaks as peaks with zero area(height)

Add the peaks with zero area(height) to calibration level

Retention Time Update:  None  Replace  Average



# PHARMA DEVILS

## QUALITY CONTROL DEPARTMENT

### STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

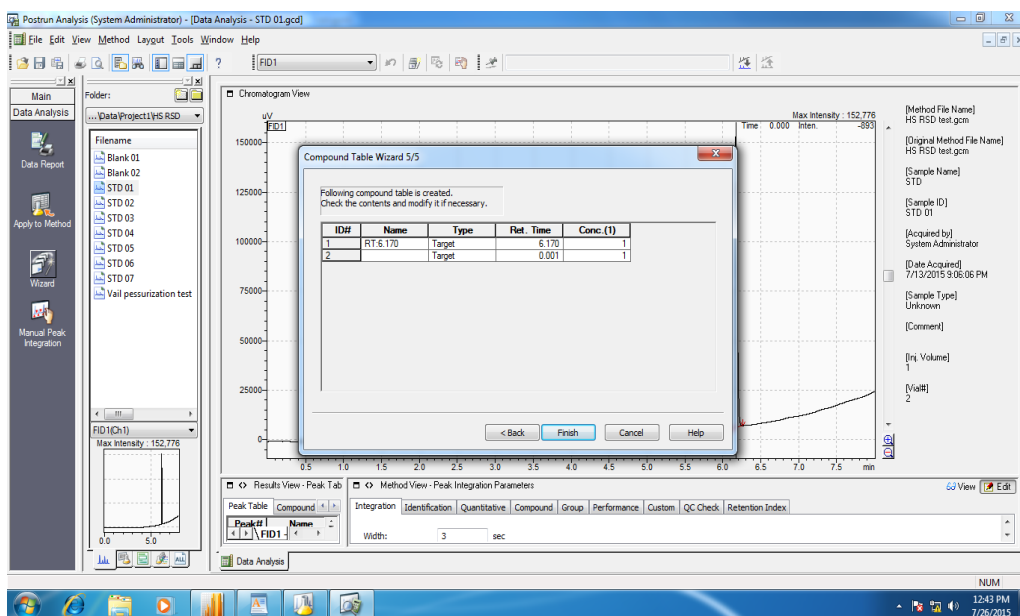
**Effective Date:**

**Supersedes:** Nil

**Review Date:**

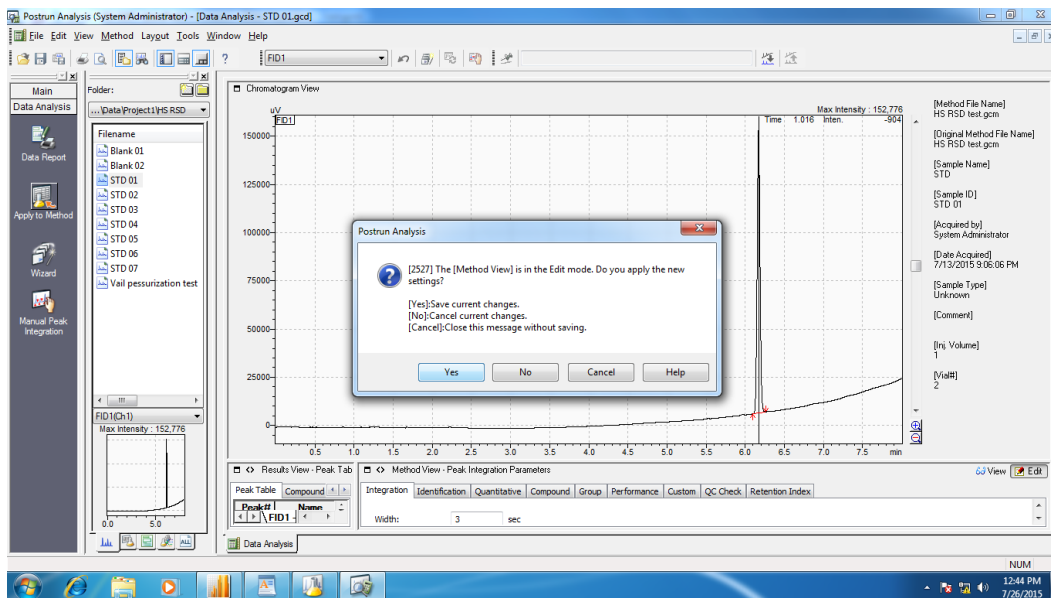
**Issue Date:**

**Page No.:**



4.1.14.22.10 Name the peak than Finished OK. The click to Apply Method.

4.1.14.22.11



4.1.14.22.12 Yes then Save the method.



# PHARMA DEVILS

## QUALITY CONTROL DEPARTMENT

### STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

**Effective Date:**

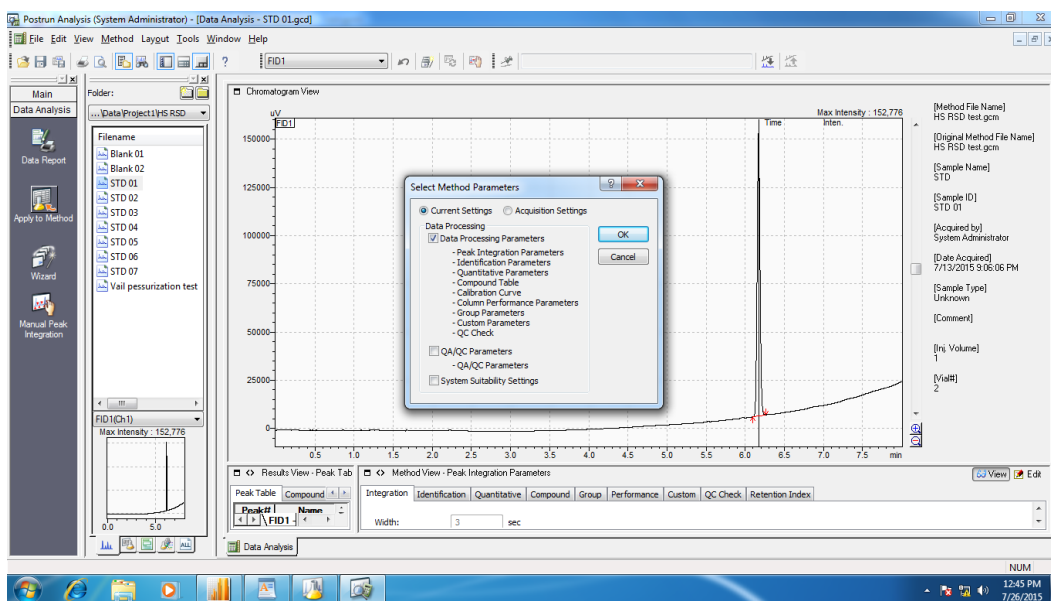
**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

4.1.14.22.13



4.1.14.22.14 Tick the QA/QC Parameter. Click Post batch select the method than possessing.

4.1.14.22.15

| Postrun | Wait | Sample Name | Sample ID | Sample Type | Method File         | Data File    | Level | Report Output | Report Format File   | Data Comment |
|---------|------|-------------|-----------|-------------|---------------------|--------------|-------|---------------|----------------------|--------------|
| 1       |      | Blank       | Blank-01  | 0:Unknown   | RSD:HS RSD test.gcm | Blank_01.gcd | 0     |               | ons\System\DEFAULT.r |              |
| 2       |      | STD         | STD 01    | 0:Unknown   | RSD:HS RSD test.gcm | STD_01.gcd   | 0     |               | ons\System\DEFAULT.r |              |
| 3       |      | STD         | STD 02    | 0:Unknown   | RSD:HS RSD test.gcm | STD_02.gcd   | 0     |               | ons\System\DEFAULT.r |              |
| 4       |      | STD         | STD 03    | 0:Unknown   | RSD:HS RSD test.gcm | STD_03.gcd   | 0     |               | ons\System\DEFAULT.r |              |
| 5       |      | STD         | STD 04    | 0:Unknown   | RSD:HS RSD test.gcm | STD_04.gcd   | 0     |               | ons\System\DEFAULT.r |              |
| 6       |      | STD         | STD 05    | 0:Unknown   | RSD:HS RSD test.gcm | STD_05.gcd   | 0     |               | ons\System\DEFAULT.r |              |
| 7       |      | STD         | STD 06    | 0:Unknown   | RSD:HS RSD test.gcm | STD_06.gcd   | 0     |               | ons\System\DEFAULT.r |              |
| 8       |      | STD         | STD 07    | 0:Unknown   | RSD:HS RSD test.gcm | STD_07.gcd   | 0     |               | ons\System\DEFAULT.r |              |
| 9       |      | Blank       | Blank-2   | 0:Unknown   | RSD:HS RSD test.gcm | Blank_02.gcd | 0     |               | ons\System\DEFAULT.r |              |

4.1.14.22.16 Open the Standard report format generated in the report format.

4.1.14.22.17 Select the data result and transfer to standard report format.

4.1.14.22.18 Display will show the standard report with chromatogram and results.

4.1.14.22.19 Click to print the report and select the printer, then click OK.

4.1.14.22.20 Record the operation details in instrument log book of GC.

**4.1.15 HS 20 Headspace Auto sampler.**



**STANDARD OPERATING PROCEDURE**

**Department:** Quality Control

**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

**Effective Date:**

**Supersedes:** Nil

**Review Date:**

**Issue Date:**

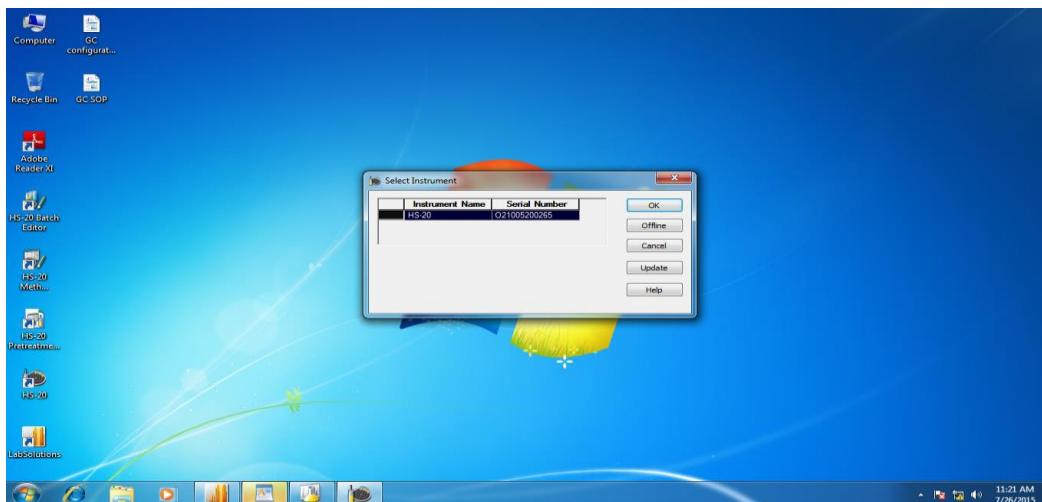
**Page No.:**

4.1.15.1 Double Click the Head Space icon.



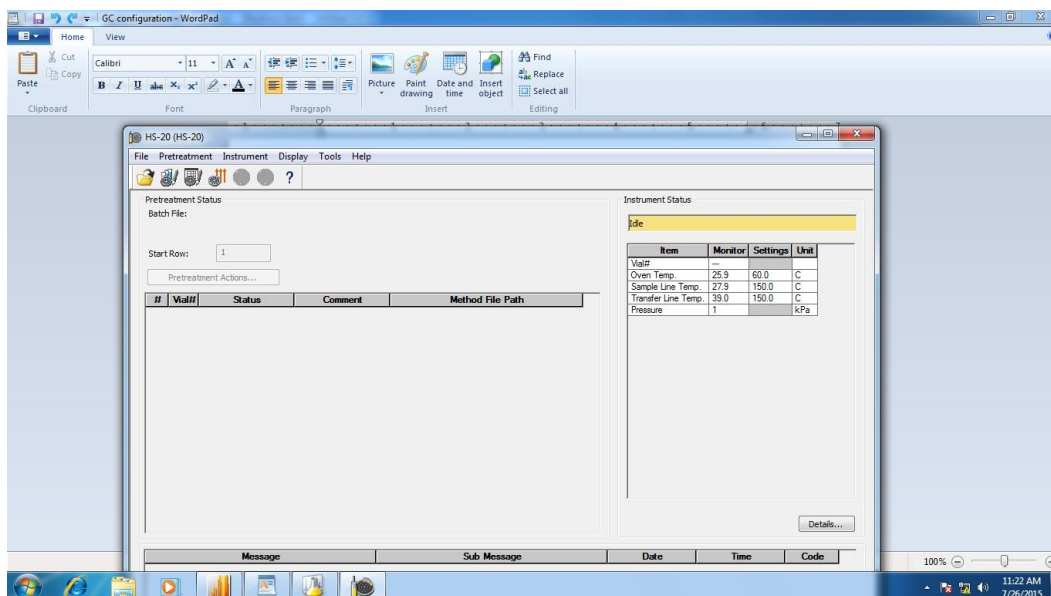
HS-20.lnk

4.1.15.2 HS-20 Program Initializing and window display on the Screen.



Than ok.

4.1.15.3



4.1.15.4 Click the instrument status Click on method edit.



## STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

**Effective Date:**

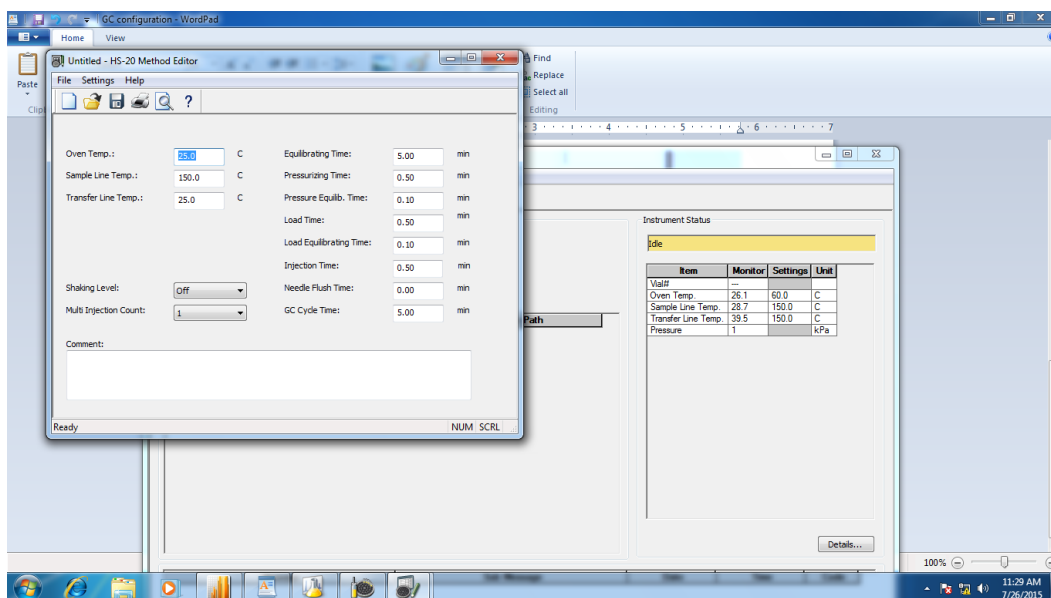
**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

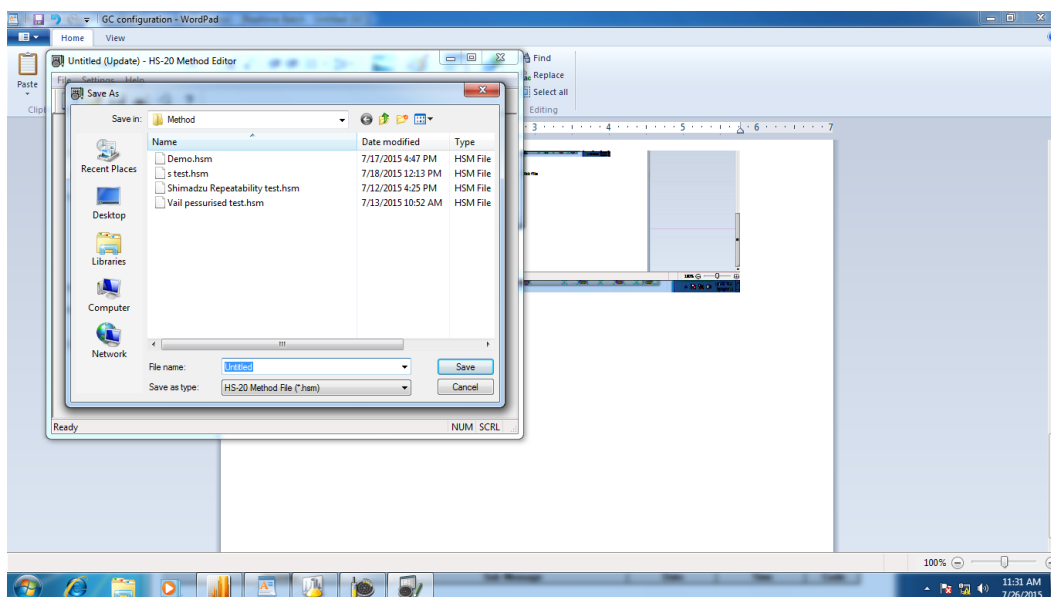
4.1.15.5



4.1.15.6

Feed the method parameter and Comment then ok. Click File and save as.

4.1.15.7



4.1.15.8

Name of method then ok.

4.1.15.9

Select the batch editor.



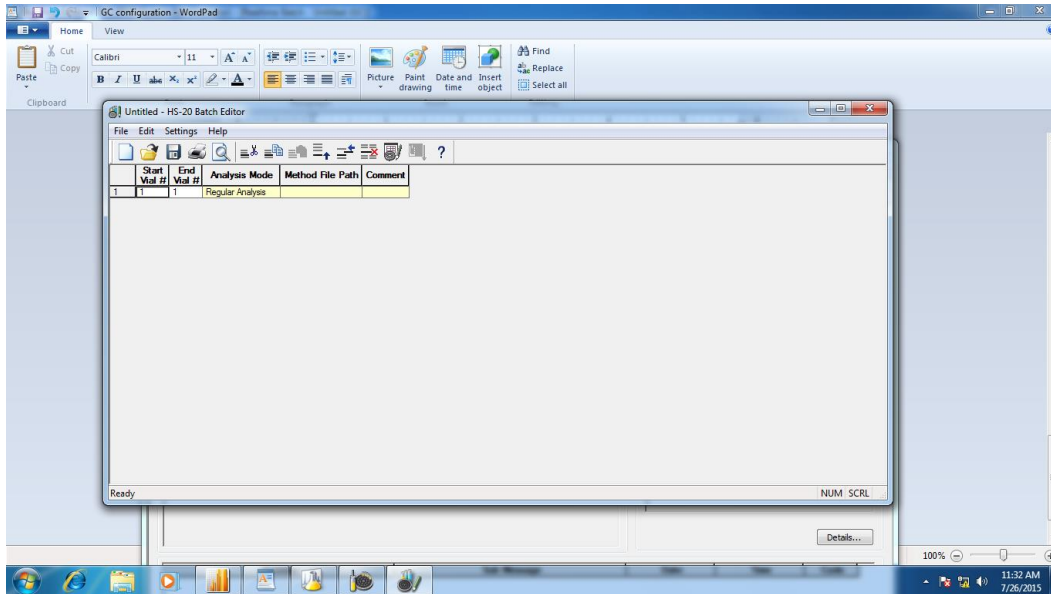
# PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

## STANDARD OPERATING PROCEDURE

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

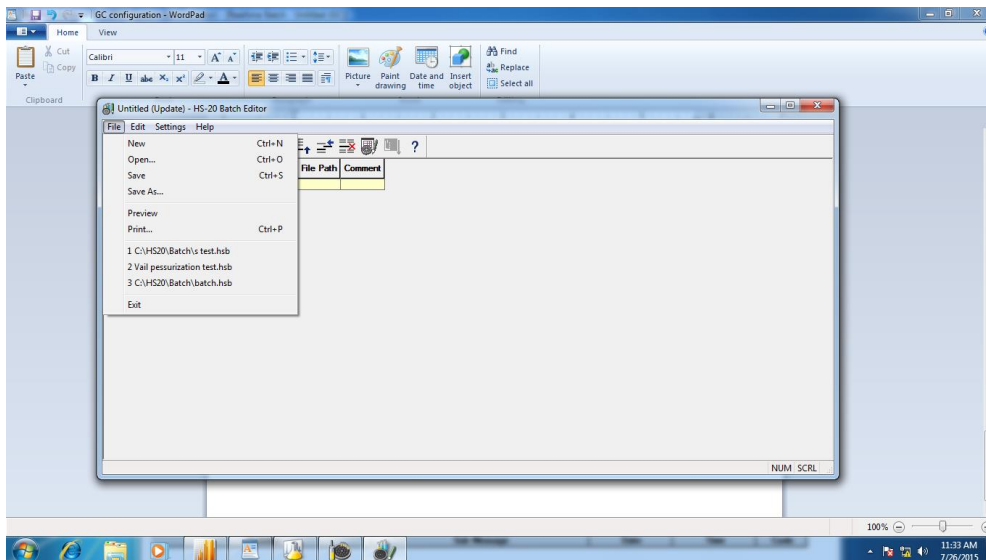
4.1.15.10



4.1.15.11

Feed the details and save the batch.

4.1.15.12





## STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

**Effective Date:**

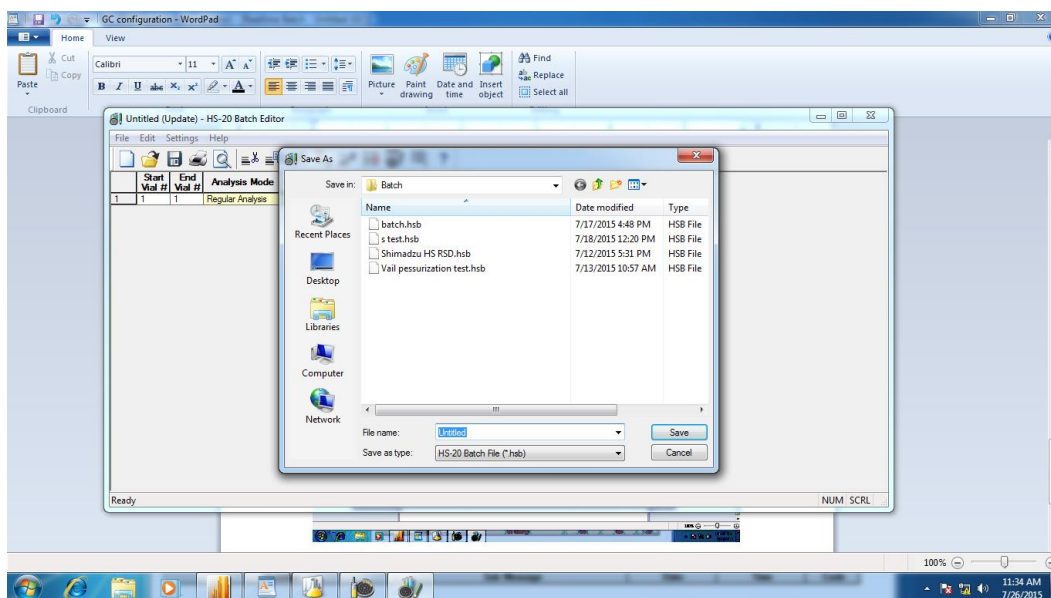
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**Review Date:**

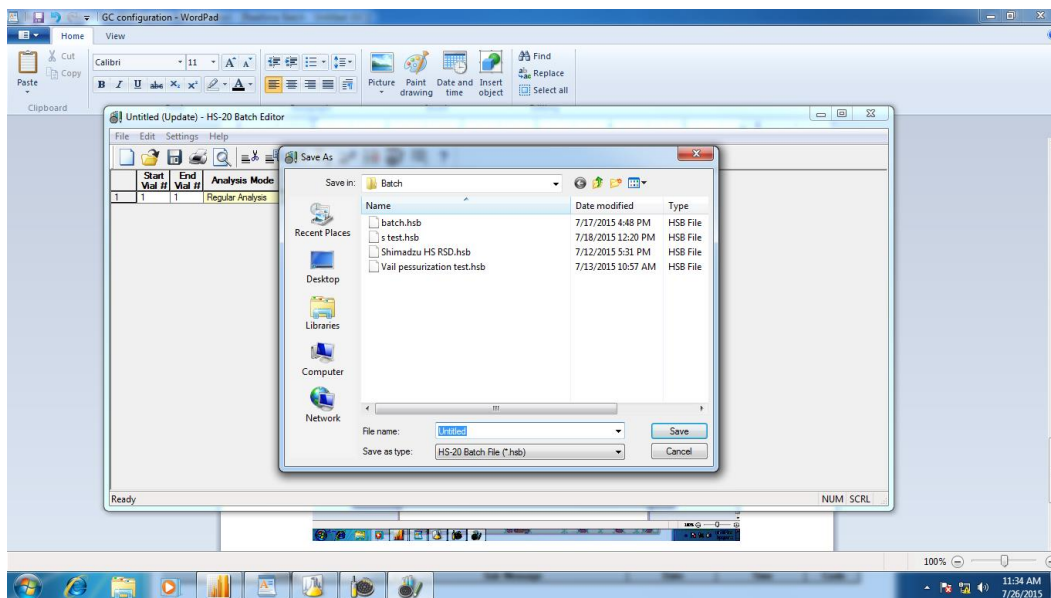
**Issue Date:**

**Page No.:**

4.1.15.13



4.1.15.14



4.1.15.15 Save it. the Sequence in Headspace.

4.1.15.16 Same Sequence Created the GC to be Start the Same time Run .But GC sequence only Generated the Data.

4.1.15.17 After completion of this take print of Method file and sequence respectiviliy.

**4.1.17 Conditioning of capillary and packed column**





**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

4.1.17.1 Carry out the conditioning of the column when impurities have adhered to the liquid phase of the capillary column or baseline becomes unstable or ghost peaks are observed in the chromatogram or peak detection of components is not obtained.

4.1.17.2 Set the flow rate of carrier gas as per the requirement of analysis.

4.1.17.3 Set the temperature of the column oven approximately 30°C higher than that is required for analysis. Do not exceed the maximum allowable temperature of the column.

4.1.17.4 Set the temperature of the detector approximately 30°C higher than that of the column oven.

4.1.17.5 Perform conditioning for two or three hours or more depending on the degree of contamination.

4.1.17.6 After completion of conditioning process of the column, ensure for the system suitability of the column as per the requirement of the product to be analysed and after obtaining satisfactory results use the column for further analysis.

**4.2 Calibration:**

**4.2.1 Flow Calibration:**

**Frequency: Quarterly**

**4.2.1.1 For capillary column:**

4.2.1.1.1 Connect the column (30 m X 0.32 mm ID, 1.8-µm film thickness) fused silica analytical column (BP 624) to the gas chromatograph.

4.2.1.1.2 Connect the flow meter to detector outlet.

4.2.1.1.3 Carry out the calibration of Gas flow keeping oven temperature, injector temperature and detector temperature at ambient.

4.2.1.1.4 Put on the carrier gas and note down three readings consecutively at frequency of one minute at set flow rate for 0.5 mL/min, 1.0 mL/min, 3.0 mL/min, 5.0 mL/min and 10.0 mL/min.

**4.2.1.1.5 Acceptance Criteria:**

4.2.1.1.5.1 % RSD of the three readings flow rate should not be more than 2.0%

4.2.1.1.5.2 Flow accuracy: ± 20 % of the set flow rate.

**4.2.2 Calibration of Head space and Auto sampler (Precision, Carry Over and Linearity of Flame Ionization & TCD Detector and Precision and Linearity of Injector):**

**4.2.2.1 Chromatographic conditions(Flam Ionization Detector):**



**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

Column : 30m X 0.32mm X 1.8  $\mu$ m fused silica analytical column (BP - 624) or equivalent.

Carrier gas : Nitrogen

Column Oven temperature : 60°C, 2 min hold, Rate: 30°C/min  $\rightarrow$  210°C, final temperature  $\rightarrow$  8 min hold.

Column flow : 2 mL/min

Injector port temperature : 240°C

Detector port temperature : 260°C

Injection Volume : 1 $\mu$ l of each solution (For Liquid injector)

Split ratio : 10

**Chromatographic conditions(TCD Detector):**

Column : 30m X 0.32mm X 1.8  $\mu$ m fused silica analytical column (BP - 624) or equivalent

Carrier gas : Nitrogen

Column Oven temperature : 40°C, 0 min hold, Rate: 25°C/min  $\rightarrow$  90°C, 170°C @ 15°C/min, Hold for 15 min .

Column flow 9.99 ml/min

Injector port temperature 250 °C

Detector port temperature 270 °C

Injection Volume 1 $\mu$ l

Split ratio 2.0

Make Up Flow (TCD) 30.0 ml/min

Run Time 22.33 min

**Head Space Conditions for Shimadzu (HS20):**

Oven Temp : 90 °C

Sample Line Temp : 100 °C

Transfer Line Temp : 95 °C

Equilibrate Time : 20 min

Pressuring Time : 1.0 min

Pressure Equilibrating Time : 1.0 min

Load time : 1.0 min



**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

Load equilibrating time : 1.0 min

Inject time : 1.0 min

Needle flush time : 1.0 min

GC Cycle Time : 25.0 min

**4.2.2.2 Prepare standard Solution as given below.**

**Preparation of Stock Solution:**

4.2.2.2.1 Stock solution (A): Weigh accurately about 1.0 g Ethanol in 100 mL volumetric flask containing about 50 mL dimethylsulfoxide. Shake the flask and dilute up to the mark with dimethylsulfoxide. Further dilute 10 mL to 100 mL with dimethylsulfoxide.

4.2.2.2.2 Stock Solution (B): Weigh accurately about 1.0 g Isopropanol in 100 mL volumetric flask containing about 50 mL of dimethylsulfoxide. Shake the flask and dilute up to the mark with dimethylsulfoxide. Further dilute 10 mL to 100 mL with dimethylsulfoxide.

**4.2.2.3 Resolution Solution:**

4.2.2.3.1 Pipette out 10 mL stock solution (A) and 10 mL of stock solution (B) in 100 mL volumetric flask and dilute up to mark with dimethylsulfoxide.

**4.2.2.4 Standard Solution: (FID DETECTOR)**

4.2.2.4.1 Level – I: Pipette out 5.0 mL of stock solution (B) in 100 mL volumetric flask and dilute up to mark with dimethylsulfoxide. This is 50 ppm solution.

4.2.2.4.2 Level – II: Pipette out 7.5 mL of stock solution (B) in 100 mL volumetric flask and dilute up to mark with dimethylsulfoxide. This is 75 ppm solution.

4.2.2.4.3 Level- III: Pipette out 10.0 mL of stock solution (B) in 100 mL volumetric flask and dilute up to mark with dimethylsulfoxide. This is 100 ppm solution.

4.2.2.4.4 Level – IV: Pipette out 12.5 mL of stock solution (B) in 100 mL volumetric flask and dilute up to mark with dimethylsulfoxide. This is 125 ppm solution.

4.2.2.4.5 Level – V: Pipette out 15.0 mL of stock solution (B) in 100 mL volumetric flask and dilute up to mark with dimethylsulfoxide. This is 150ppm solution.

**4.2.2.4.6 Standard Solution : (TCD DETECTOR)**

4.2.2.4.6.1 weigh about 33.0 mg of each standard of n- Tetradecane, n- Pentadecane, n- Hexadecane in 100 ml Volumetric flask containing 20 ml of n- hexane and make up the volume with n- Hexane. And prepare the solution as below.



**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

- 4.2.2.4.6.2 Inject 1 $\mu$ l, of above standard six times.
- 4.2.2.5 Inject a resolution solution and record resolution between ethanol and Isopropanol.
- 4.2.2.6 Carry out three determinations of each level and record area response and retention time of Isopropanol.
- 4.2.2.7 For carry over of liquid injections Inject 1  $\mu$ L dimethylsulfoxide after triplicate injection of Level - V (150 ppm) and record the chromatogram.
- 4.2.2.8 Inject 0.5 $\mu$ L, 1.0 $\mu$ L, 2.0 $\mu$ L, standard solutions (100 ppm) in triplicate, record area response and retention time of Isopropanol for liquid injector.
- 4.2.2.9 For Headspace sampler, take 2 mL from each above solutions in three HS vials. Load the program as per conditions mentioned under 4.2.2.1 and inject the sample and evaluate the chromatograms.
- 4.2.2.10 For carry over of headspace sampler take 2 mL dimethylsulfoxide in HS vial, inject after triplicate injection of Level - V (150 ppm) and record the chromatogram.
- 4.2.2.11 Acceptance Criteria for Auto sampler(FID & TCD):**
- 4.2.2.11.1 The resolution between the peaks of ethanol and Isopropanol obtained in the chromatogram of resolution solution should not be less than 2.0.
- 4.2.2.11.2 The RSD of retention time of Isopropanol for each level should not be more than 2.0%.
- 4.2.2.11.3 The RSD of peak area response of Isopropanol for each level should not be more than 5.0%.
- 4.2.2.11.4 Plot the linearity graph of mean peak area against the concentration (ppm) of Isopropanol. The correlation coefficient should not be less than 0.99.
- 4.2.2.11.5 Carry over of Isopropanol is not more than 1% of third injection area of 150 ppm solution.
- 4.2.2.11.6 The RSD of retention time of Isopropanol for each injection volume should not be more than 2.0%.
- 4.2.2.11.7 The RSD of peak area response of Isopropanol for each injection volume should not be more than 5.0%.
- 4.2.2.11.8 Plot the linearity graph of different injection volume concentration against peak responses. The correlation coefficient,  $r^2$  value should not be less than 0.99.
- 4.2.2.11.9 RSD for each solvent peak areas of six injections should not be more than 5.0 %. For TCD.
- 4.2.2.11.10 The RSD of retention time of for each solvent should not be more than 2.0%.



**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

**4.2.2.12 Acceptance Criteria for Head Space:**

- 4.2.2.12.1 The resolution between the peaks of ethanol and Isopropanol obtained in the chromatogram of resolution solution should not be less than 2.0.
- 4.2.2.12.2 The RSD of retention time of Isopropanol should not be more than 2.0%.
- 4.2.2.12.3 The RSD of peak area response of Isopropanol should not be more than 15.0%.
- 4.2.2.12.4 Plot the linearity graph of mean peak area against the concentration (ppm) of Isopropanol. The correlation coefficient should not be less than 0.99.
- 4.2.2.12.5 Carry over of Isopropanol is not more than 1% of third injection area of 150 ppm solution.

**4.2.3 Column Thermostat Calibration:**

**4.2.3.1 Isothermal:**

- 4.2.3.1.1 Place the temperature probe of the calibrated thermometer in the column oven and close the compartment.
- 4.2.3.1.2 Set the temperature to 30° C. Allow the system to equilibrate.
- 4.2.3.1.3 After 30 minutes, note down the value displayed on the calibrated thermometer.
- 4.2.3.1.4 Similarly set the temperature to 100°C, 150°C, 200°C, 250°C and 300° C note down the value.

**4.2.3.1.5 Acceptance Criteria:**

- For 30 ° C:  $\pm 3.2^{\circ}\text{C}$
- For 100 ° C and 150 ° C:  $\pm 4.2^{\circ}\text{C}$
- For 200 ° C ,250° C and 300 ° C:  $\pm 5.5^{\circ}\text{C}$

**4.2.3.2 Programming**

- 4.2.3.2.1 Place the temperature probe of the calibrated thermometer in the column seat of the column thermostat and close the compartment.
- 4.2.3.2.2 Set the column temperature as 50°C for 1 min. Raise the temperature to 62°C at the rate 12°C/min. After one minute note down the value displayed on the calibrated thermometer.

**4.2.3.3 Acceptance Criteria:**

- 4.2.3.3.1 + 5.0°C of set temperature

**4.2.4 Frequency:** Half Yearly.

- 4.2.5 After the calibration is completed, make the entries in the Annexure - I and enter the status of the calibration in the Instrument Calibration Tag.



**PHARMA DEVILS**  
QUALITY CONTROL DEPARTMENT

**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

4.2.6 If instrument is out of calibration, affix “UNDER MAINTENANCE “or “DO NOT USE” label on the instrument and immediately inform to maintenance  
And call service engineer. Follow the procedure as per out of calibration SOP.

**5.0 ANNEXURE (S):**

Annexure - I: Calibration Record of Gas chromatograph flow and Column Oven Temperature.  
Annexure - II: Calibration Record of Gas chromatograph.  
Annexure - III: Log book of Gas Chromatograph

**6.0 REFERENCE (S):**

SOP: Handling of Out of Calibration instrument.  
SOP: Preparation, approval, distribution, control, revision and destruction of Standard Operating Procedure (SOP).

**7.0 ABBREVIATION (S)/DEFINITION (S):**

SOP - Standard Operating Procedure  
RSD - Relative standard deviation  
GC - Gas Chromatography  
ppm - Parts Per Million  
°C - Degree Centigrade  
IPA - Isopropanol.  
Min. - minute

**REVISION CARD**

| S.No. | REVISION No. | REVISION DATE | DETAILS OF REVISION | REASON(S) FOR REVISION | REFERENCE CHANGE CONTROL No. |
|-------|--------------|---------------|---------------------|------------------------|------------------------------|
| 1     | 00           | ---           | ---                 | New SOP                | -                            |



**PHARMA DEVILS**  
QUALITY CONTROL DEPARTMENT

**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

**ANNEXURE I**

|                                                                                 |  |                          |                          |
|---------------------------------------------------------------------------------|--|--------------------------|--------------------------|
| <b>CALIBRATION RECORD OF GAS CHROMATOGRAPH FLOW AND COLUMN OVEN TEMPERATURE</b> |  |                          |                          |
|                                                                                 |  |                          | <b>Reference SOP No.</b> |
| <b>Location</b>                                                                 |  | <b>Model No.</b>         |                          |
| <b>Manufactured By</b>                                                          |  | <b>Instrument ID No.</b> |                          |
| <b>Date of Calibration</b>                                                      |  | <b>Frequency</b>         |                          |
| <b>Next Calibration Due on</b>                                                  |  | <b>Page No.</b>          | <b>1 of 3</b>            |

| Column Type                          |                  | Flow Rate<br>0.5 (ml)<br>(± 20 %) | Flow Rate<br>1.0 (ml)<br>(± 20 %) | Flow Rate<br>3.0 (ml)<br>(± 20 %)   | Flow Rate<br>5.0 (ml)<br>(± 20 %) | Flow Rate<br>10.0 (ml)<br>(± 20 %) | Remark                                 |
|--------------------------------------|------------------|-----------------------------------|-----------------------------------|-------------------------------------|-----------------------------------|------------------------------------|----------------------------------------|
| Capillary Column                     | Obs. Flow (mL)   |                                   |                                   |                                     |                                   |                                    | Meets / Does not meet the requirement. |
|                                      | % RSD (NMT 2.0%) |                                   |                                   |                                     |                                   |                                    |                                        |
| Test Name                            | Observation      |                                   |                                   | Acceptance Criteria                 |                                   |                                    | Remark                                 |
| <b>Column Thermostat Temperature</b> | Isothermal:      |                                   |                                   | For 30°C: ± 3.2 °C                  |                                   |                                    | Meets / Does not meet the requirement. |
|                                      | 30°C = _____     |                                   |                                   | For 100°C & 150°C: ± 4.2 °C         |                                   |                                    |                                        |
|                                      | 100° C = _____   |                                   |                                   | For 200°C, 250° C & 300°C: ± 5.5 °C |                                   |                                    |                                        |
|                                      | 150° C = _____   |                                   |                                   |                                     |                                   |                                    |                                        |
|                                      | 200° C = _____   |                                   |                                   |                                     |                                   |                                    |                                        |
|                                      | 250° C = _____   |                                   |                                   |                                     |                                   |                                    |                                        |
| 300° C = _____                       |                  |                                   |                                   |                                     |                                   |                                    |                                        |



**PHARMA DEVILS**  
QUALITY CONTROL DEPARTMENT

**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

|  |                                                                                               |                    |                                        |
|--|-----------------------------------------------------------------------------------------------|--------------------|----------------------------------------|
|  | Programming:<br>Initial temperature: 50°C = _____<br>Final Temperature: 62°C in 1 min = _____ | ± 5°C of set Temp. | Meets / Does not meet the requirement. |
|--|-----------------------------------------------------------------------------------------------|--------------------|----------------------------------------|

|                       |                    |                     |
|-----------------------|--------------------|---------------------|
| <b>Calibrated By:</b> | <b>Checked By:</b> | <b>Approved By:</b> |
| <b>Date</b> :         | <b>Date</b> :      | <b>Date</b> :       |





**PHARMA DEVILS**  
QUALITY CONTROL DEPARTMENT

**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

**1) Flow Calibration:**

**Flow meter ID:** \_\_\_\_\_ **Calibration valid Up To:** \_\_\_\_\_

**Capillary Column ID No.:** \_\_\_\_\_

| Column Type-Capillary Column |                         |                               |   |   |                    |                                                      |                             |
|------------------------------|-------------------------|-------------------------------|---|---|--------------------|------------------------------------------------------|-----------------------------|
| S.No.                        | Set flow rate (ml/ min) | Observed flow rate in ml/ min |   |   |                    | Flow rate Limits in ml/min ( $\pm 20\%$ of set flow) | (% ) RSD [Limit: NMT 2.0 %] |
|                              |                         | 1                             | 2 | 3 | Mean               |                                                      |                             |
| 1.                           | 0.5                     |                               |   |   |                    | 0.4 to 0.6                                           |                             |
| 2.                           | 1.0                     |                               |   |   |                    | 0.8 to 1.2                                           |                             |
| 3.                           | 3.0                     |                               |   |   |                    | 2.4 to 3.6                                           |                             |
| 4.                           | 5.0                     |                               |   |   |                    | 4.0 to 6.0                                           |                             |
| 5.                           | 10.0                    |                               |   |   |                    | 8.0 to 12.0                                          |                             |
| <b>Performed By:</b>         |                         |                               |   |   | <b>Checked By:</b> |                                                      |                             |



**PHARMA DEVILS**  
QUALITY CONTROL DEPARTMENT

**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

**2) Column Thermostat Calibration:**

**Thermometer ID:** \_\_\_\_\_ **Calibration valid up to:** \_\_\_\_\_

|             | Temperature (°C)                  | Observed Temperature (°C) | Limit                 |
|-------------|-----------------------------------|---------------------------|-----------------------|
| Isothermal  | 30° C                             |                           | 26.8°C to 33.2°C      |
|             | 100° C                            |                           | 95.8°C to 104.2°C     |
|             | 150° C                            |                           | 145.8°C to 154.2°C    |
|             | 200° C                            |                           | 194.5°C to 205.5°C    |
|             | 250° C                            |                           | 244.5°C to 255.5°C    |
|             | 300° C                            |                           | 294.5°C to 305.5°C    |
| Programming | Initial Temperature - 50°C        |                           | 45°C to 55°C          |
|             | Final Temperature - 62°C in 1 min |                           | 57°C to 67°C in 1 min |

Result: Complies / Does not comply

|                      |                    |
|----------------------|--------------------|
| <b>Performed By:</b> | <b>Checked By:</b> |
|----------------------|--------------------|

**Remark:** The Instrument Calibration is **OK/ Not OK** as per **In-House** requirements.

|                       |                    |                     |
|-----------------------|--------------------|---------------------|
| <b>Calibrated By:</b> | <b>Checked By:</b> | <b>Approved By:</b> |
| <b>Date</b> :         | <b>Date</b> :      | <b>Date</b> :       |



**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

**ANNEXURE II**

**CALIBRATION RECORD OF GAS CHROMATOGRAPH**

|                                                |  |                                |                      |
|------------------------------------------------|--|--------------------------------|----------------------|
| <b>CALIBRATION RECORD OF GAS CHROMATOGRAPH</b> |  |                                |                      |
|                                                |  |                                | <b>Reference SOP</b> |
| <b>Location</b>                                |  | <b>Model No.</b>               |                      |
| <b>Manufactured By</b>                         |  | <b>Instrument ID No.</b>       |                      |
| <b>Date of Calibration</b>                     |  | <b>Next Calibration Due on</b> |                      |
| <b>Frequency</b>                               |  | <b>Page No. :</b>              | <b>35 of 8</b>       |

**Calibration Summary Sheet**

| Test Name                                                                                      | Observation                                                |         |           |             | Acceptance Criteria                                    | Remark                                 |
|------------------------------------------------------------------------------------------------|------------------------------------------------------------|---------|-----------|-------------|--------------------------------------------------------|----------------------------------------|
| <b>Auto sampler Precision, Carry Over and Linearity of FID &amp; TCD detector and injector</b> | <b>Precision, Carry Over and Linearity of FID detector</b> | Conc.   | RSD of RT | RSD of Area | RSD of RT :<br>NMT 2.0 %<br>RSD of Area :<br>NMT 5.0 % | Meets / Does not meet the requirement. |
|                                                                                                |                                                            | 50 ppm  |           |             |                                                        |                                        |
|                                                                                                |                                                            | 75 ppm  |           |             |                                                        |                                        |
|                                                                                                |                                                            | 100 ppm |           |             |                                                        |                                        |
|                                                                                                |                                                            | 125 ppm |           |             |                                                        |                                        |
|                                                                                                |                                                            | 150 ppm |           |             |                                                        |                                        |
|                                                                                                | Correlation coefficient 'r' =                              |         |           |             | NLT 0.99                                               |                                        |
|                                                                                                | Carryover=                                                 |         |           |             | NMT 1.0%                                               |                                        |
|                                                                                                | Resolution =                                               |         |           |             | NLT 2.0                                                |                                        |
|                                                                                                | <b>Precision, of TCD detector.</b>                         | Solvent | RSD of RT | RSD of Area | RSD of RT :<br>NMT 2.0 %<br>RSD of Area :<br>NMT 5.0 % | Meets / Does not meet the requirement. |
| Tetradecane                                                                                    |                                                            |         |           |             |                                                        |                                        |
| Pentadecane                                                                                    |                                                            |         |           |             |                                                        |                                        |
| Hexadecane                                                                                     |                                                            |         |           |             |                                                        |                                        |



# PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

## STANDARD OPERATING PROCEDURE

**Department:** Quality Control

**SOP No.:**

**Title:** Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space)

**Effective Date:**

**Supersedes:** Nil

**Review Date:**

**Issue Date:**

**Page No.:**

| Precision,<br>and<br>Linearity of<br>injector | Injection<br>volume | RSD of<br>RT | RSD of<br>Area | RSD of RT :<br>NMT 2.0 % | Meets / Does<br>not meet the<br>requirement. |
|-----------------------------------------------|---------------------|--------------|----------------|--------------------------|----------------------------------------------|
|                                               | 0.5 $\mu$ L         |              |                | RSD of Area              |                                              |
|                                               | 1.0 $\mu$ L         |              |                | :                        |                                              |
|                                               | 2.0 $\mu$ L         |              |                | NMT 5.0 %                |                                              |
| Correlation coefficient 'r' =                 |                     |              |                | NLT 0.99                 |                                              |



**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

| <b>Head Space sampler Linearity, Precision, Carry Over and Linearity of detector:</b> | Conc.                         | RSD of RT | RSD of Area | Meets/ does not meet the requirement. |          |
|---------------------------------------------------------------------------------------|-------------------------------|-----------|-------------|---------------------------------------|----------|
|                                                                                       | 50 ppm                        |           |             |                                       |          |
|                                                                                       | 75 ppm                        |           |             |                                       |          |
|                                                                                       | 100 ppm                       |           |             |                                       |          |
|                                                                                       | 125 ppm                       |           |             |                                       |          |
|                                                                                       | 150 ppm                       |           |             |                                       |          |
|                                                                                       | Correlation coefficient 'r' = |           |             |                                       | NLT 0.99 |
|                                                                                       | Carryover =                   |           |             |                                       | NMT 1.0% |
| Resolution =                                                                          |                               |           | NLT 2.0     |                                       |          |

|                       |                    |                     |
|-----------------------|--------------------|---------------------|
| <b>Calibrated By:</b> | <b>Checked By:</b> | <b>Approved By:</b> |
| <b>Date :</b>         | <b>Date :</b>      | <b>Date :</b>       |

**1.0 Calibration of Head Space Sampler & Auto sampler (Precision, Carry Over and Linearity of Flame Ionization Detector and Precision and Linearity of Injector.**

**Balance ID: EQ/QCD/**

Standard Preparation:

Stock solution (A):

Weigh accurately \_\_ g (about 1.0 g) Ethanol in \_\_ mL (100 mL) volumetric flask containing \_\_\_\_ mL (about 50 mL) dimethylsulfoxide. Shake the flask and dilute up to the mark with dimethylsulfoxide. Dilute \_\_\_\_ mL (10 mL) to \_\_\_\_ mL (100 mL) with dimethylsulfoxide.

Stock solution (B):

Weigh accurately \_\_ g (about 1.0 g) Isopropanol in \_\_\_\_ mL (100 mL) volumetric flask containing \_\_\_\_ mL (about 50 mL) of dimethylsulfoxide. Shake the flask and dilute up to the mark with water. Dilute \_\_\_\_ mL (10 mL) to \_\_\_\_ mL (100) with dimethylsulfoxide.

**Resolution Solution:**

Pipette out \_\_\_\_ ml (10 ml) stock solution (A) and \_\_\_\_ ml (10 ml) of stock solution (B) in \_\_\_\_ ml (100 ml) volumetric flask and dilute up to mark with dimethylsulfoxide.

Standard Solution:

**Level-I:** Pipette out \_\_\_\_ ml (5.0 ml) of stock solution (B) in \_\_\_\_ ml (100 ml) volumetric flask and dilute up to mark with dimethylsulfoxide. (50 ppm solution).



**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

**Level-II:** Pipette out \_\_\_\_\_ ml (7.5 ml) of stock solution (B) in \_\_\_\_\_ ml (100 ml) volumetric flask and dilute up to mark with dimethylsulfoxide. (75 ppm solution).

**Level-III:** Pipette out \_\_\_\_\_ ml (10.0ml) of stock solution (B) in \_\_\_\_\_ ml (100 ml) volumetric flask and dilute up to mark with dimethylsulfoxide. (100 ppm solution).

**Level-IV:** Pipette out \_\_\_\_\_ ml (12.5 ml) of stock solution (B) in \_\_\_\_\_ ml (100 ml) volumetric flask and dilute up to mark with dimethylsulfoxide. (125 ppm solution).

**Level-V:** Pipette out \_\_\_\_\_ ml (15.0 ml) of stock solution (B) in \_\_\_\_\_ ml (100 ml) volumetric flask and dilute up to mark with dimethylsulfoxide. (150 ppm solution).

**Chromatographic conditions for Liquid Auto Sampler:**

Column : BP-624 (30m X 0.32mm X 1.8  $\mu$ m fused silica analytical column)

Column ID : \_\_\_\_\_

Carrier gas: Nitrogen : \_\_\_\_\_

Column Oven temperature : 60°C, 2 min hold, Rate: 30°C/min  $\rightarrow$  210°C, final temperature  $\rightarrow$  8 min hold.

Column flow : 2 ml/min : \_\_\_\_\_

Injector port temperature : 240°C : \_\_\_\_\_

Detector port temperature: 260°C : \_\_\_\_\_

Injection Volume : 1 $\mu$ l : \_\_\_\_\_

Detector attn. : 1 : \_\_\_\_\_

Split ratio : 10 : \_\_\_\_\_

**System Suitability:**

- The resolution between the peaks of ethanol and Isopropanol obtained in the chromatogram of resolution solution is \_\_\_\_\_ (Not less than 2.0).



**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

**1.1 Precision, Carry Over and Linearity of Flame Ionization Detector:**

| S.No | CONCENTRATION |      |          |      |          |      |          |      |          |      |
|------|---------------|------|----------|------|----------|------|----------|------|----------|------|
|      | 50 PPM        |      | 75 PPM   |      | 100PPM   |      | 125PPM   |      | 150 PPM  |      |
|      | RT (min)      | Area | RT (min) | Area | RT (min) | Area | RT (min) | Area | RT (min) | Area |
| 1.   |               |      |          |      |          |      |          |      |          |      |
| 2.   |               |      |          |      |          |      |          |      |          |      |
| 3.   |               |      |          |      |          |      |          |      |          |      |
| Mean |               |      |          |      |          |      |          |      |          |      |
| S.D  |               |      |          |      |          |      |          |      |          |      |
| %RSD |               |      |          |      |          |      |          |      |          |      |

RSD limit (For Area) : Not more than 5.0 %

RSD limit (For RT (Retention time)): Not more than 2.0 %

Complies / Does not comply

**Performed By:**

**Checked By:**

**Auto samplers carry over:**

| Peak area of IPA in Blank | Peak area of IPA in sample | Calculation formula                                           | % Carry over | Acceptance Criteria |
|---------------------------|----------------------------|---------------------------------------------------------------|--------------|---------------------|
|                           |                            | Peak area of IPA in blank x 100<br>Peak area of IPA in sample |              | NMT : 1%            |

Complies / Does not comply

**Performed By:**

**Checked By:**

**1.2 Precision and Linearity of Injector:**

| Injection Volume (µL) | Injection No. | 1 | 2 | 3 | Mean | % RSD | Limits                                                                      |
|-----------------------|---------------|---|---|---|------|-------|-----------------------------------------------------------------------------|
| 0.5                   | RT (Min)      |   |   |   |      |       | RSD limit (For Area) :<br>Not more than 5.0 %<br><br>RSD limit (For R.T.) : |
|                       | Area          |   |   |   |      |       |                                                                             |
| 1.0                   | RT (Min)      |   |   |   |      |       |                                                                             |
|                       | Area          |   |   |   |      |       |                                                                             |
| 2.0                   | RT (Min)      |   |   |   |      |       |                                                                             |



**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

|  |      |  |  |  |  |  |                     |
|--|------|--|--|--|--|--|---------------------|
|  | Area |  |  |  |  |  | Not more than 2.0 % |
|--|------|--|--|--|--|--|---------------------|

|                      |                    |
|----------------------|--------------------|
| <b>Performed By:</b> | <b>Checked By:</b> |
|----------------------|--------------------|

**1.3 Precision of TCD Detector:**

**Standard Preparation:**

Weigh about \_\_\_\_\_(33.0 mg ) of each standard of n- Tetradecane, n- Pentadecane, n- Hexadecane in 100 ml Volumetric flask containing 20 ml of n- hexane and make up the volume with n- Hexane.

**Chromatographic conditions for Liquid Auto Sampler:**

Column : BP-624 (30m X 0.32mm X 1.8 µm fused silica analytical column)

Column ID: \_\_\_\_\_

Carrier gas: Nitrogen: \_\_\_\_\_

Column Oven temperature : 40°C, 0 min hold, Rate: 25°C/min → 90°C,170°C @ 15°C/min, Hold for 15 min .

Column flow : 9.99mL/min : \_\_\_\_\_

Injector port temperature: 250°C : \_\_\_\_\_

Detector port temperature: 270°C : \_\_\_\_\_

Injection Volume : 1µl : \_\_\_\_\_

Make Up Flow : 30.0 ml/min : \_\_\_\_\_

Run Time : 22.33 min : \_\_\_\_\_

|   | SOLVENT       |      |               |      |              |      |
|---|---------------|------|---------------|------|--------------|------|
|   | N-Tetradecane |      | N-Pentadecane |      | N-Hexadecane |      |
|   | RT (min)      | Area | RT (min)      | Area | RT (min)     | Area |
| 1 |               |      |               |      |              |      |
| 2 |               |      |               |      |              |      |
| 3 |               |      |               |      |              |      |
| 4 |               |      |               |      |              |      |
| 5 |               |      |               |      |              |      |







**PHARMA DEVILS**  
QUALITY CONTROL DEPARTMENT

**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
|------------------------------------------------------------------------------------------------------------|------------------------|
| <b>Department:</b> Quality Control                                                                         | <b>SOP No.:</b>        |
| <b>Title:</b> Operation and Calibration of Gas Chromatograph (Shimadzu GC-2010 Plus with HS 20 Head Space) | <b>Effective Date:</b> |
| <b>Supersedes:</b> Nil                                                                                     | <b>Review Date:</b>    |
| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

RSD limit (For Area) : Not more than 5.0 %

RSD limit (For RT (Retention time)): Not more than 2.0 %

Complies / Does not comply



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QUALITY CONTROL DEPARTMENT

**STANDARD OPERATING PROCEDURE**

|                                                                                                            |                        |
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| <b>Issue Date:</b>                                                                                         | <b>Page No.:</b>       |

**Head Space sampler carry over:**

| Peak area of IPA in Blank  | Peak area of IPA in sample | Calculation formula                                                                     | % Carry over       | Acceptance Criteria |
|----------------------------|----------------------------|-----------------------------------------------------------------------------------------|--------------------|---------------------|
|                            |                            | $\frac{\text{Peak area of IPA in blank} \times 100}{\text{Peak area of IPA in sample}}$ |                    | NMT : 1.0%          |
| Complies / Does not comply |                            |                                                                                         |                    |                     |
| <b>Performed By:</b>       |                            |                                                                                         | <b>Checked By:</b> |                     |

**Remark:** The Instrument Calibration is **OK/ Not OK** as per **In-House** requirements.

|                       |                    |                     |
|-----------------------|--------------------|---------------------|
| <b>Calibrated By:</b> | <b>Checked By:</b> | <b>Approved By:</b> |
| <b>Date</b> :         | <b>Date</b> :      | <b>Date</b> :       |

