When Gas Leaks Cannot Be Stopped

Cause	Solution	See also
Part is incorrectly installed.	Install part correctly.	-
Graphite ferrule or other seal such as O-ring is damaged.	• Replace O-ring. • Additionally tighten the graphite ferrule. If there is leakage even after additional tightening, replace it.	Maintenance Help
Tubing including pipes and joints is damaged.	Replacement of the tubing part is required. Turn off the main power of the instrument and contact your Shimadzu sales/service representative.	-

■ Carrier gas leak check (Capillary)

Gas leak causes poor repeatability. It also wastes the carrier gas. Check that there is no carrier gas leak.

1 Prepare the instrument.

- When the GC operates

1 Press (HOME) - [GC Start/Stop Sequence] to display [GC Stop Sequence] screen.

2 Set [Flow Control] at [On].

3 Press [Stop GC].

- When the GC stops

1 Press (HOME) - [GC Start/Stop Sequence] to display [GC Start Sequence] screen.

2 Set [Start Temp/Det] at [Disable].

3 Press [Start GC].

2 Press Monitor (Monitor).

Monitor screen appears.

3 Wait until the temperatures of the injection port and the detector drops below 50 °C.

4 Ensure that the column oven temperature drops below 40 °C.

5 Press the icon of the Injection port.

[INJ/FLOW] screen appears.

6 Turn [Off] the flow controller control.

7 Set [Purge Flow] at "0" mL/min.

8 Open the column oven door and remove the column on the inlet side.

9 Seal the connection of the column in the injection port.

10 Install a blank nut (G-type (P/N: S221-35566-92)) and a column gasket (P/N: S201-35184) on the purge vent. For SPL or PTV, install a blank nut also on the split vent.

11 Ensure that actual value of [Carrier Gas Primary Pressure] is above "300 kPa".

12 Set parameters at the following values.

Injection Mode	:	Split
Control Mode	:	Press
Inlet Press	:	300 kPa
Total Flow	:	100 mL/min

13 Turn [On] the flow controller control.

14 Wait until [Inlet Press] increases to around carrier gas primary pressure, and then turn [Off] the flow controller control.

15 Record actual value of [Inlet Press].

16 Wait about 10 minutes and check the decrease of the value. Compare it with actual value of the inlet pressure recorded in step 15.

The followings indicate the presence of a leak.

Actual value of [Inlet Press] in step 15	Leak judgment
Below 300 kPa	The decrease is more than 2.5 kPa
300 to 450 kPa	The decrease is more than 5.0 kPa
450 to 600 kPa	The decrease is more than 7.5 kPa
600 to 750 kPa	The decrease is more than 10.0 kPa
Above 750 kPa	The decrease is more than 12.5 kPa

17 If gas leak is found, resolve it.

Solutions for gas leaks

18 Return the connection and settings to original state.

Set [Start Temp/Det] on [GC Start Sequence] screen at [Enable] to start temperature control after you return the settings.

■ Carrier gas leak check (Packed)

Gas leak causes poor repeatability. It also wastes the carrier gas. Check that there is no carrier gas leak.

1 Prepare the instrument.

- When the GC operates

1 Press (HOME) - [GC Start/Stop Sequence] to display [GC Stop Sequence] screen.

2 Set [Flow Control] at [On].

3 Press [Stop GC].

- When the GC stops

1 Press (HOME) - [GC Start/Stop Sequence] to display [GC Start Sequence] screen.

2 Set [Start Temp/Det] at [Disable].

3 Press [Start GC].

2 Press Monitor (Monitor).

Monitor screen appears.

3 Wait until the temperatures of the injection port and the detector drops below 50 °C.

4 Ensure that the column oven temperature drops below 40 °C.

5 Press the icon of the Injection port.

[INJ/FLOW] screen appears.

6 Turn [Off] the flow controller control.

7 Open the column oven door and remove the column on the inlet side.

> **Note**
> When removing the column, pull the adapter straight down to avoid breaking the tip of the glass insert.

8 Seal the connection of the column in the injection port.

9 Set [Column Flow] at "100" mL/min.

10 Turn [On] the flow controller control.

11 Wait until [Inlet Press] increases to around carrier gas primary pressure, and then turn [Off] the flow controller control.

12 Record actual value of [Inlet Press].

13 Wait about 10 minutes and check the decrease of the value. Compare it with actual

value of the inlet pressure recorded in step 12.

The followings indicate the presence of a leak.

Actual value of [Inlet Press] in step 12	Leak judgment
Below 300 kPa	The decrease is more than 2.5 kPa
300 to 450 kPa	The decrease is more than 5.0 kPa
450 to 600 kPa	The decrease is more than 7.5 kPa
600 to 750 kPa	The decrease is more than 10.0 kPa
Above 750 kPa	The decrease is more than 12.5 kPa

14 If gas leak is found, resolve it.

Solutions for gas leaks

15 Return the connection and settings to original state.

Set [Start Temp/Det] on [GC Start Sequence] screen at [Enable] to start temperature control after you return the settings.

Solutions for gas leaks

Check whether the following parts have gas leaks using leak detector or Snoop.

- Septum
- Around the injection port
- Connections of tubing
- Split vent (Around the blank nut)
- Purge vent (Around the blank nut)
- Column connection

WARNING

Instructions

Take precautions when using Snoop or similar soap solution not to drip onto electronic parts or wiring.

This may cause electric shock.

CAUTION

Prohibitions

Do not use the leak detecting fluid nor soapy water for gas leak check on the connections above the carrier and detector gas controllers (AFC/APC).

The drips may damage the controller.

If the leak position is found, perform the following actions.

See Maintenance Help for the replacement procedure of the parts.

Item	Corrective action
Septum	Replace the septum.

Around the injection port	Replace O-ring of the glass insert.
Split vent	Replace the seal of blank nut.
Purge vent	Replace blank nut.
Column connection	Replace ferrule, column nut, or ClickTek connector (optional).

Note

- In some sensitive analyses, Snoop can interfere with proper detection. In those situations, use a leak detector.
- The instrument allows 0.2 mL/min leakage from split/purge vent.

[INJ/FLOW] Screen (SPL)

Press (HOME) - [Injection Port] to display [INJ/FLOW] screen.

The screen is different depending on the injection units configured in the analytical line.

When several injection units are installed on the instrument and are configured in several analytical lines, press [LINE] in the top-right of the screen to switch lines and make settings.

This section describes the screen for SPL.

No.	Item	Description

<table>
<tr><td>❶</td><td>Control of Flow Controller</td><td>Displays the current control status of the flow controller. Press [Off] or [On] to switch the control status.
Set at [On] to control the flow controller in the injection port. When you press [Start GC] on [GC Start Sequence] screen, the instrument starts to control the injection port which is configured in the analytical line and whose ❶ [Control of Flow Controller] is set at [On]. For the injection port configured in the analytical line, ❷ [Inj Temp] can be controlled even when ❶ [Control of Flow Controller] is set at [Off].
Note
If this function is turned [Off] while the instrument performs analysis or column temperature is high, it stops supply of the carrier gas and may result in column degradation due to oxidization of the liquid phase.
<table><tr><td>Items</td><td>Off, On</td></tr><tr><td>Default</td><td>On</td></tr></table></td></tr>
<tr><td>❷</td><td>Inj Temp</td><td>Sets the injection port temperature. Select a temperature suitable for each sample to instantly vaporize the sample in the injection port.
Note
・ The use at high temperature may reduce the service life of the temperature sensor. Do not increase the temperature higher than necessary.
・ Due to vapor pressure of the sample elements, the sample also vaporizes at temperatures lower than the boiling point of sample elements.
<table><tr><td>Range</td><td>0.0 to 450.0 °C</td></tr><tr><td>Default</td><td>25.0 °C</td></tr></table></td></tr>
<tr><td>❸</td><td>Inlet Press</td><td>Sets the inlet pressure of the column. Set initial pressure when you use Column Inlet Press Program.
<table><tr><td>Range</td><td>0.0 to 970.0 kPa</td></tr><tr><td>Default</td><td>100.0 kPa</td></tr></table></td></tr>
<tr><td>❹</td><td>Col Flow</td><td>Sets column flow. The instrument calculates the column inlet pressure based on inner diameter, length, and film thickness of the column, which are set on [Column Information] screen to achieve the set column flow at the initial temperature in the column oven temperature program.
<table><tr><td>Range</td><td>• When ⓫ [Carrier Gas Type] is He, N2, or Ar
0.00 to 1300.00 mL/min
• When ⓫ [Carrier Gas Type] is H2
0.00 to 50.00 mL/min</td></tr><tr><td>Default</td><td>1.00 mL/min</td></tr></table></td></tr>
</table>

<table>
<tr><td>❺</td><td>Linear Vel</td><td colspan="2">Sets the average linear velocity of the carrier gas flowing in the capillary column. The instrument calculates the column inlet pressure based on inner diameter, length, and film thickness of the column, which are set on [Column Information] screen so that the gas flows in the column at the set velocity at the initial temperature in the column oven temperature program.</td></tr>
<tr><td></td><td></td><td>Range</td><td>0.0 to 99999.9 cm/s</td></tr>
<tr><td></td><td></td><td>Default</td><td>40.0 cm/s</td></tr>
<tr><td>❻</td><td>Split Ratio</td><td colspan="2">The split ratio is "split flow / column flow".
When you set a split ratio, the instrument sets the total flow so that the desired split ratio occurs at the column oven temperature. Set the split ratio to [-1.0] to fix the total flow regardless of the column oven temperature.</td></tr>
<tr><td></td><td></td><td>Range</td><td>-1.0, 0.0 to 9999.9</td></tr>
<tr><td></td><td></td><td>Default</td><td>-1.0</td></tr>
<tr><td>❼</td><td>Total Flow</td><td colspan="2">Sets the total flow, which is the sum total of column flow, split flow, and septum purge flow.
Reference AFC and APC Control Ranges</td></tr>
<tr><td></td><td></td><td>Range</td><td>0.0 to 1300.0 mL/min</td></tr>
<tr><td></td><td></td><td>Default</td><td>50.0 mL/min</td></tr>
<tr><td>❽</td><td>Injection Mode</td><td colspan="2">Sets injection mode.</td></tr>
<tr><td></td><td></td><td>Items</td><td>• Split
Controls the column inlet pressure and the total flow so that the column inlet pressure and split ratio occur as specified.
• Splitless
Closes the split flow line during the sampling time and controls the Total Flow Controller (TFC) so that the column inlet pressure remains the set value.
Opens the split flow line after the sampling time elapses and controls the Electronic Split Controller (ESC) so that the column inlet pressure remains the set value.</td></tr>
<tr><td></td><td></td><td>Default</td><td>Split</td></tr>
<tr><td>❾</td><td>Sampling time</td><td colspan="2">Sets the sampling time for splitless analysis The sampling time indicates the period of time after analysis starts until the split flow line is opened. This item is displayed when ❽ [Injection Mode] is set at [Splitless].
Note
For the correct functioning of the sampling time, ensure that the sampling time is shorter than the hold time of the column initial temperature.</td></tr>
<tr><td></td><td></td><td>Range</td><td>0.00 to 9999.99 min</td></tr>
</table>

		Default	1.00 min
⑩	Flow Control Mode	Sets flow control mode.	
		Items	• Press Controls the instrument so that the inlet pressure remains constant during analysis. • Velocity Controls the instrument so that the velocity remains constant during analysis. • Col Flow Controls the instrument so that the column flow remains constant during analysis.
		Default	Velocity
⑪	Carrier Gas Type	Specify the carrier gas type supplied to the AFC. This parameter is used for the calculation of pressure, column flow, and velocity. **Note** If this parameter is not set correctly, the calculations may not be performed correctly. For example, if this parameter is set at "N2" when "He" is actually used, the displayed pressure, column flow, velocity, and total flow are not correct.	
		Items	He, N2, H2, Ar
		Default	He
⑫	Primary Press	A rough pressure level (primary pressure) of the carrier gas supplied to the AFC is displayed. **Note** It is normal differences among equipment that primary pressures displayed on the screen for each injection port have approximately 10 kPa variation when two or more injection ports are mounted.	
⑬	Purge Flow	Sets septum purge flow. This is the initial flow in the purge flow program.	
		Range	0.0 to 1300.0 mL/min **Note** Control range is different depending on the column inlet pressure. Reference AFC and APC Control Ranges
		Default	3.0 mL/min
⑭	Press Program	Displays [Column Inlet Press Program] screen. This item is displayed when ⑩ [Flow Control Mode] is set at [Press]. Reference [Column Inlet Press Program] Screen	
	Linear Vel Program	Displays [Linear Velocity Program] screen. This item is displayed when ⑩ [Flow Control Mode] is set at [Velocity]. Reference [Linear Velocity Program] Screen	

	Col Flow Program	Displays [Column Flow Program] screen. This item is displayed when ⑩ [Flow Control Mode] is set at [Col Flow]. Reference [Column Flow Program] Screen
⑮	Back Flush Setting	Displays [Back Flush Settings] screen. This item is displayed when AFT and the injection port are configured in the same analytical line. Reference [Back Flush Settings] Screen

Submenu

Item	Description	See also
Column Information	Displays [Column Information] screen.	[Column Information] Screen
Carrier Gas Saver	Displays [Carrier Gas Saver] screen. The carrier gas saver saves carrier gas by reducing the split flow.	[Carrier Gas Saver] Screen
Split Ratio Program	Displays [Split Ratio Program] screen. Split ratio can be changed during analysis.	[Split Ratio Program] Screen
Purge Flow Program	Displays [Purge Flow Program] screen. Sets septum purge program.	[Purge Flow Program] Screen
High Press Setting	Displays [High Pressure Injection] screen. The instrument keeps the column inlet pressure at high value for a certain period of time during the injection.	[High Pressure Injection] Screen
Splitter Hold	Displays [Splitter Hold] screen. The instrument fixes the split flow line and keep the split ratio constant.	[Splitter Hold] Screen
Calibration Offset	Press [Run] to perform calibration so that the displayed AFC pressure becomes "0 kPa" at atmospheric pressure. Use this function before GC starts or if the displayed pressure is not "0 kPa" while the gas supply stops (exposed to atmospheric pressure).	Offset Calibration