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LabSolutions

Instruction Manual

AFT (Advanced Flow Technology) Guide

Read this manual thoroughly before you use the product.
Keep this manual for future reference.

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Introduction

Read this Instruction Manual thoroughly before using the product.

Thank you for purchasing this product.

This manual describes how to use this product. Read this manual thoroughly before using the product and operate the product in accordance with the instructions in this manual.

Keep this manual for future reference.

IMPORTANT

- If the user or usage location changes, ensure that this Instruction Manual is always kept together with the product.
- If this manual or a product warning label is lost or damaged, immediately contact your Shimadzu representative to request a replacement.
- To ensure safe operation, contact your Shimadzu representative if product installation, adjustment, re-installation (after the product is moved), or repair is required.

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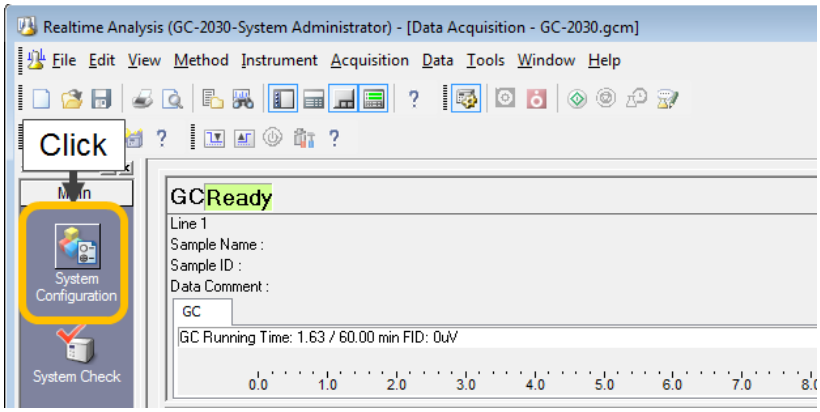
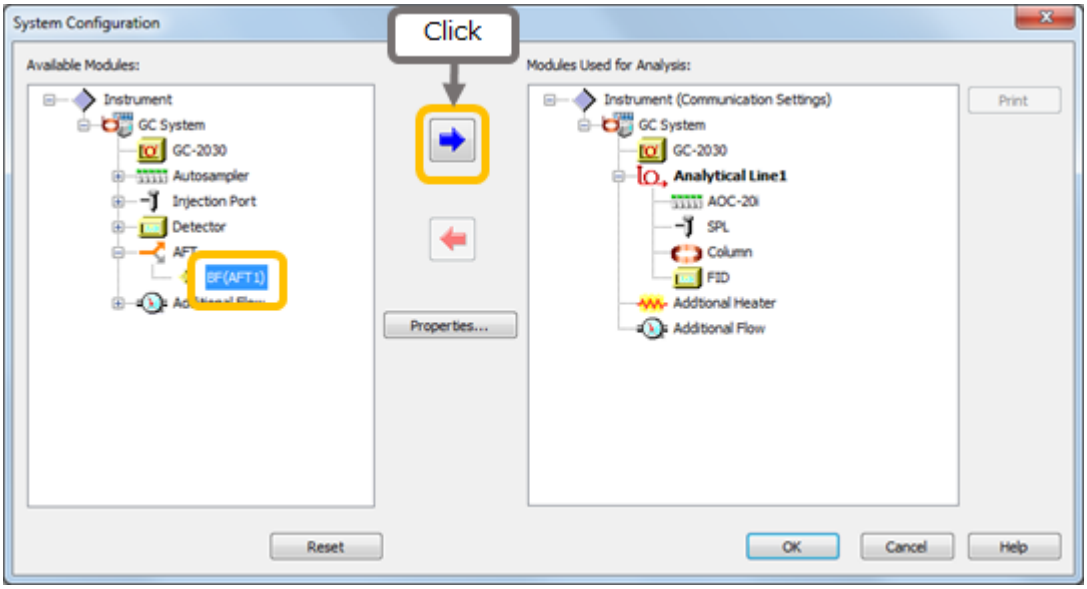
1.1 Settings for Analytical Line configuration

1.1.1 Setting Procedure

1 **Display the [System Configuration] window.**



2 **Select the AFT that are used at [Available Modules], click , and move them to [Modules Used for Analysis].**



NOTE When using Heart-Cut or Detector Switching, set it at "Analytical Line1".

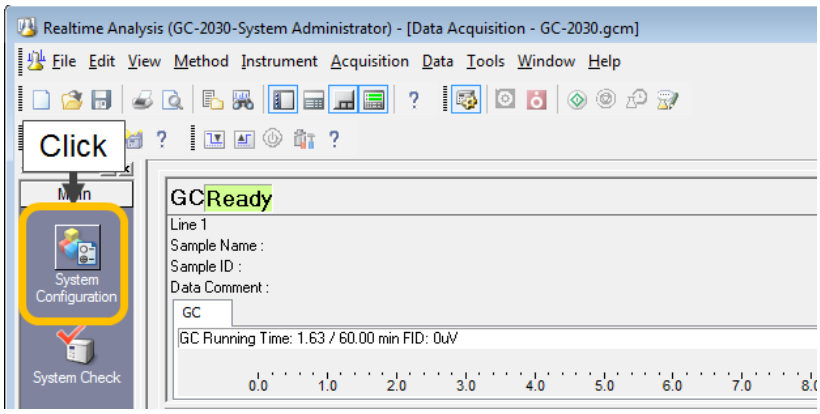
3 **Click [OK] to apply the system configuration settings.**

1.2 Settings for Restrictors

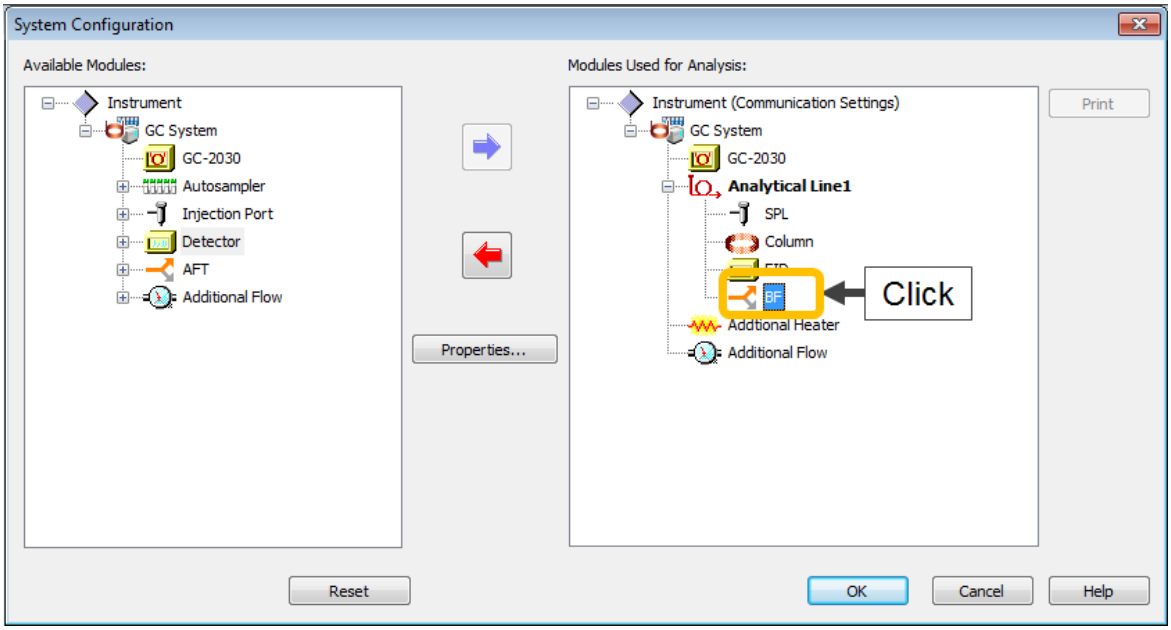
For Advanced Flow Technology (AFT) systems, use restrictors adjusted to match each of the systems. This section describes, using the back flush system as an example, how to set the inner diameter and the length of a restrictor with LabSolutions when the restrictor is installed.

1.2.1 Setting Procedure

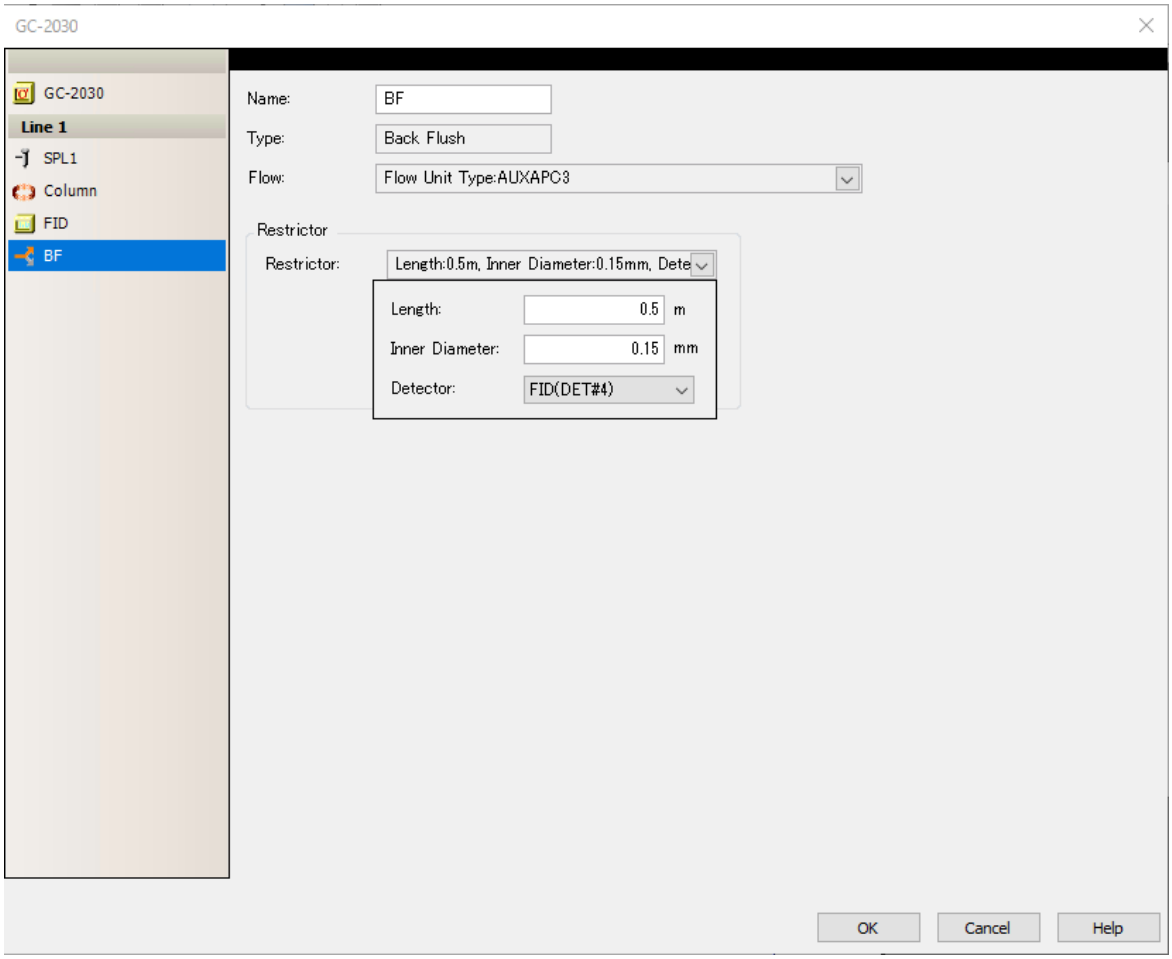
1 **Display the [System Configuration] window.**



2 **Click [AFT] in the [System Configuration] window.**



3 Configure the parameters in the [Restrictor] area.



NOTE In [Detector], select the detector to which the restricor is connected.

4 Click [OK] to apply the system configuration settings.

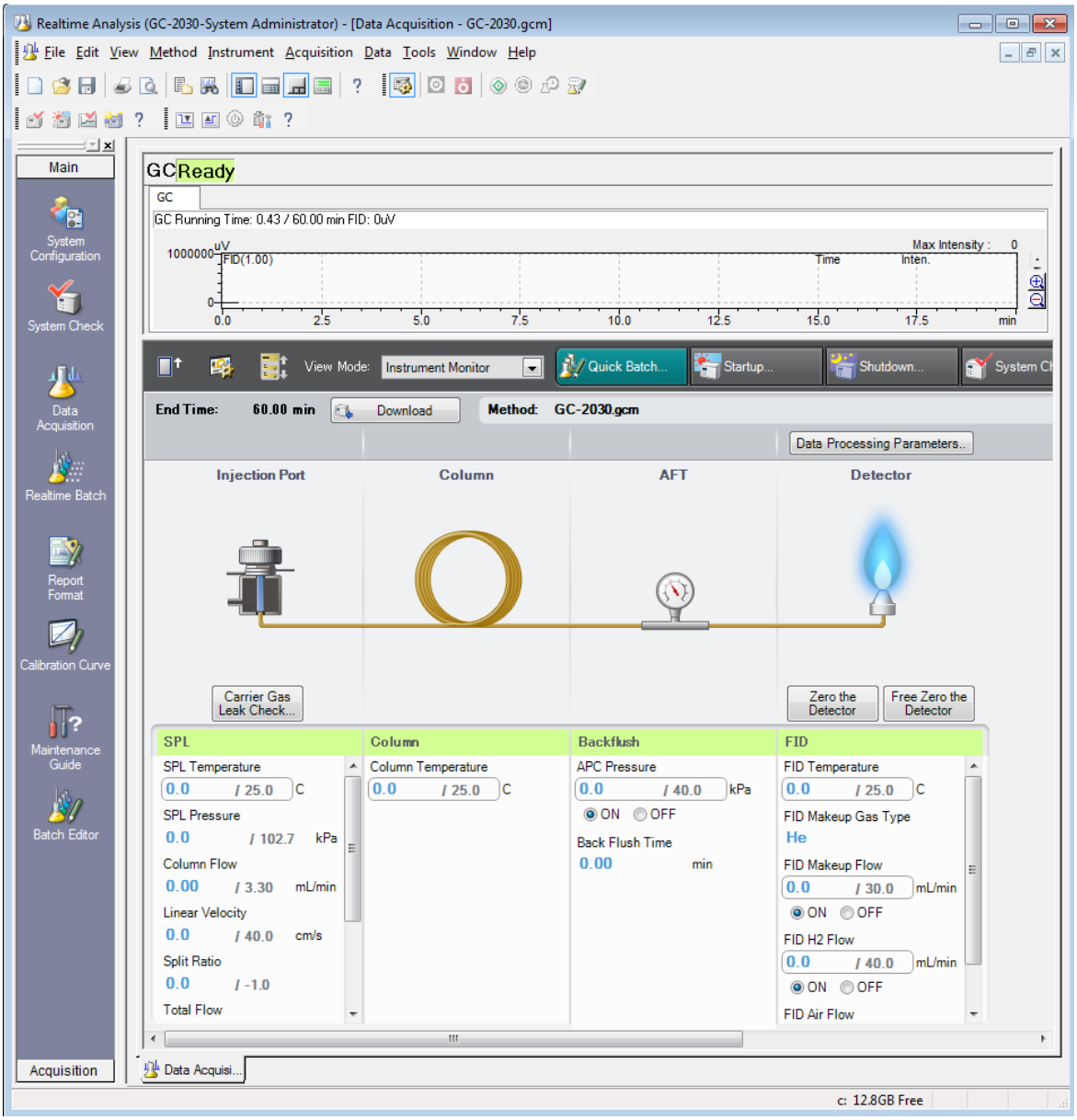
2 Back Flush

2.1 What Is the Back Flush Feature?

The back flush system expels substances remaining inside a column from the inlet by reversing the flow of the carrier gas after the target compounds have been detected. This can heighten productivity by shortening analysis times. In addition, it can lessen column contamination by effectively expelling high boiling point substances, which may cause column contamination, from the split vent.

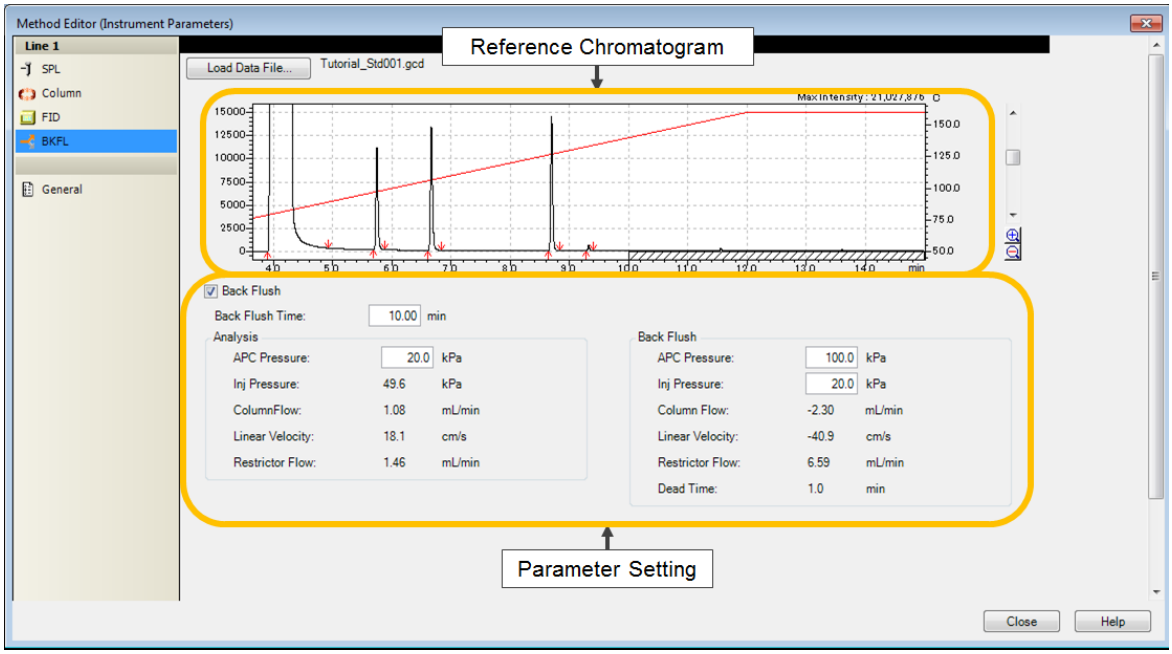
The back flush element is attached to the outlet of an ordinary analysis column. This controls the analysis column outlet pressure (hereinafter referred to as APC pressure), enabling the carrier gas to flow during back flush in the direction opposite to that during ordinary analysis. For connection between the element and the detector, a restrictor with an inner diameter of 0.15 mm and a length of 0.5 m is used as standard. The APC pressure is ordinarily maintained at about 50 kPa, even when back flush is not being performed. Accordingly, in analyses using the back flush element, the column linear velocity and flowrate are determined based on the difference between the ordinary column inlet pressure and the APC pressure. In the LabSolutions [Method Editor (Instrument Parameters)] window, it is possible to configure and display the accurate column linear velocity and split ratio with consideration to these points. It is also possible

to confirm the carrier gas flowrate as it flows in the opposite direction during back flush. This makes it easy to develop a method for performing a back flush.



2.2 Editing an Instrument Method

2.2.1 [Method Editor (Instrument Parameters)] Window

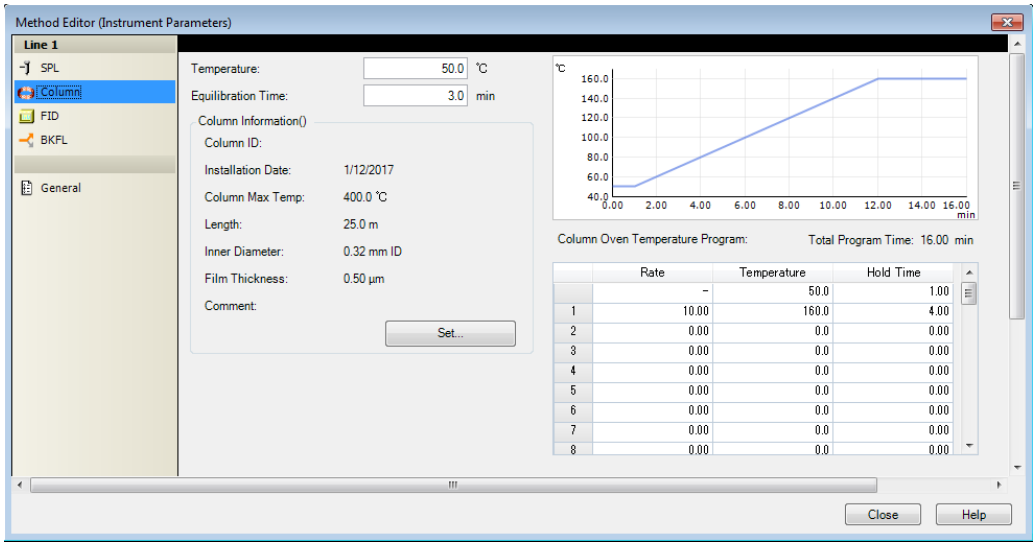


Name	Explanation	Remarks
[Load Data File] button	Click this button to load data files to be used as a reference when setting the time to start a back flush.	
[Back Flush] checkbox	Select this checkbox when performing a back flush. Back flush will not be performed if you perform an analysis using a method with this checkbox cleared.	
[Back Flush Time]	Double-click the reference chromatogram display area to set the back flush start time.	
([Analysis] area) [APC Pressure]	Configure the advanced pressure controller (APC) pressure (column outlet pressure) when back flush is not being performed.	Be aware that setting the APC pressure too high may have an adverse impact, such as extinguishing the flame in the FID detector.
([Back Flush] area) [APC Pressure] / [Inj Pressure]	Configure the APC pressure and injector pressure during a back flush.	Be aware that setting the sample injector pressure too low may prevent the purge flow.

2.2.2 Setting Procedure

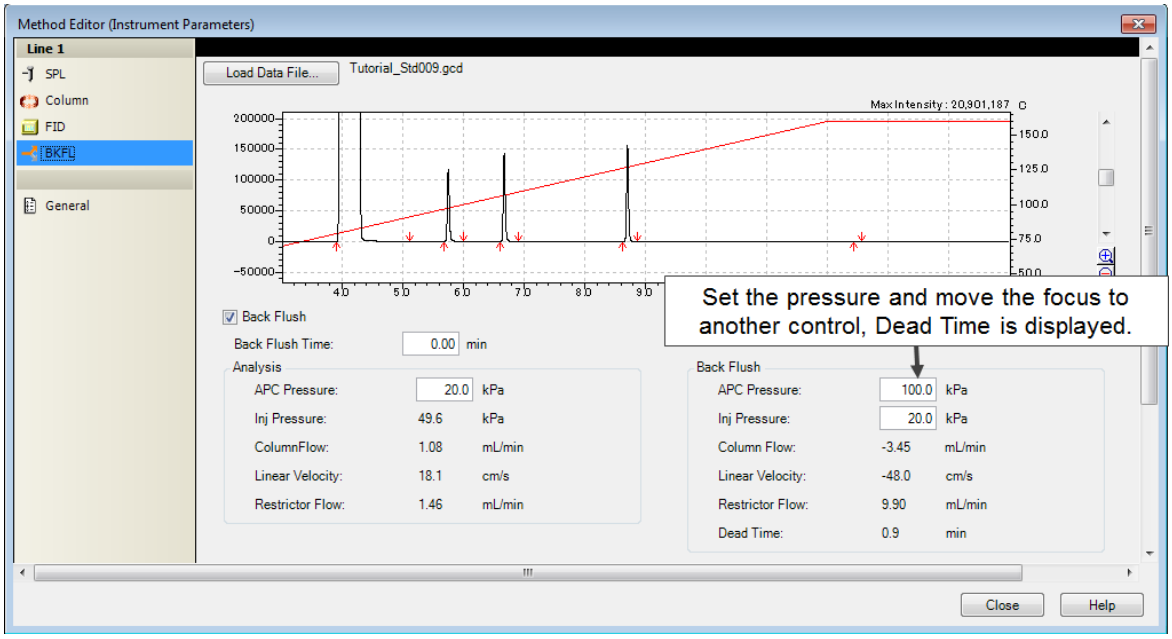
1 **Perform an analysis using ordinary conditions, in which a back flush is not performed.**

1. Clear the [Back Flush] checkbox.
2. Configure the other analysis conditions and perform the analysis.



2 **Click [Load Data File] to load the file of data acquired from the analysis in step 1.**

3 Set [APC Pressure] and [Inj Pressure] in the [Back Flush] area.

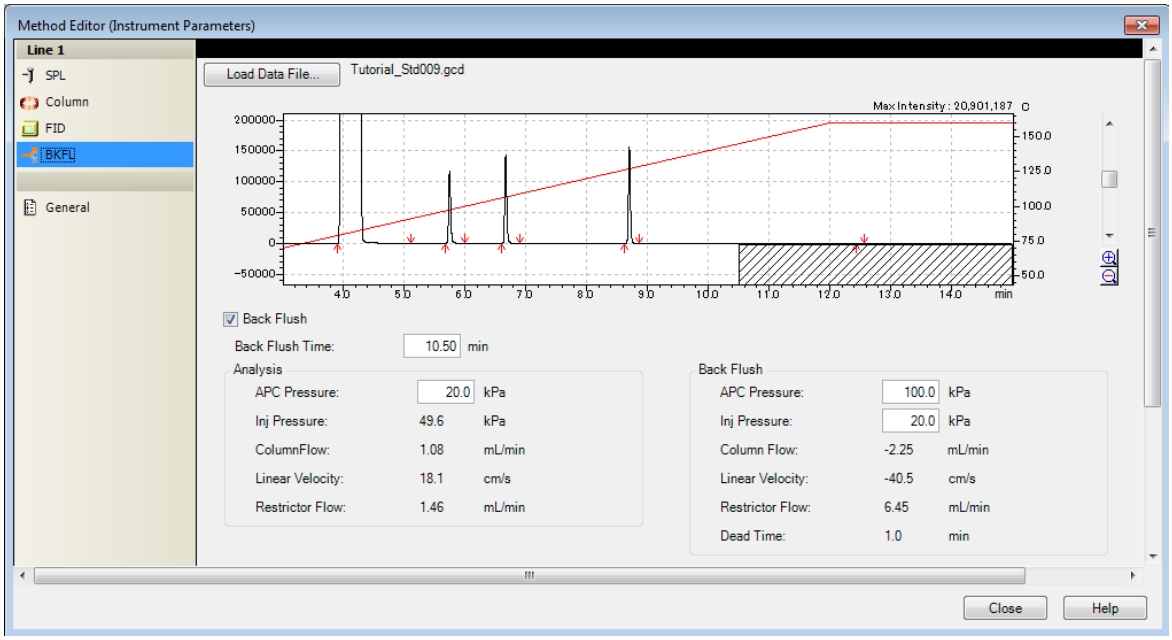


Hint Dead time refers to the time required for the carrier gas to travel from the splitter to the injector. Ordinarily, substances remaining inside the column will be discharged within about 1.5 times this dead time. Use this as a guide when developing methods. In this case, enter the temperature during the back flush as the column temperature. Caution is necessary because if the back flush starts while the temperature is rising, the column flowrate will be reduced as the column temperature rises. Accordingly, all of the substances remaining in the column may not be discharged unless the back flush time is set to longer than 1.5 times the dead time.

NOTE

- Setting the value in [APC Pressure} too high may have an adverse impact, such as extinguishing the flame in the flame ionization detector.
- Setting the value in [Inj Pressure] too low may prevent the purge flow. Ordinarily, use the default value.

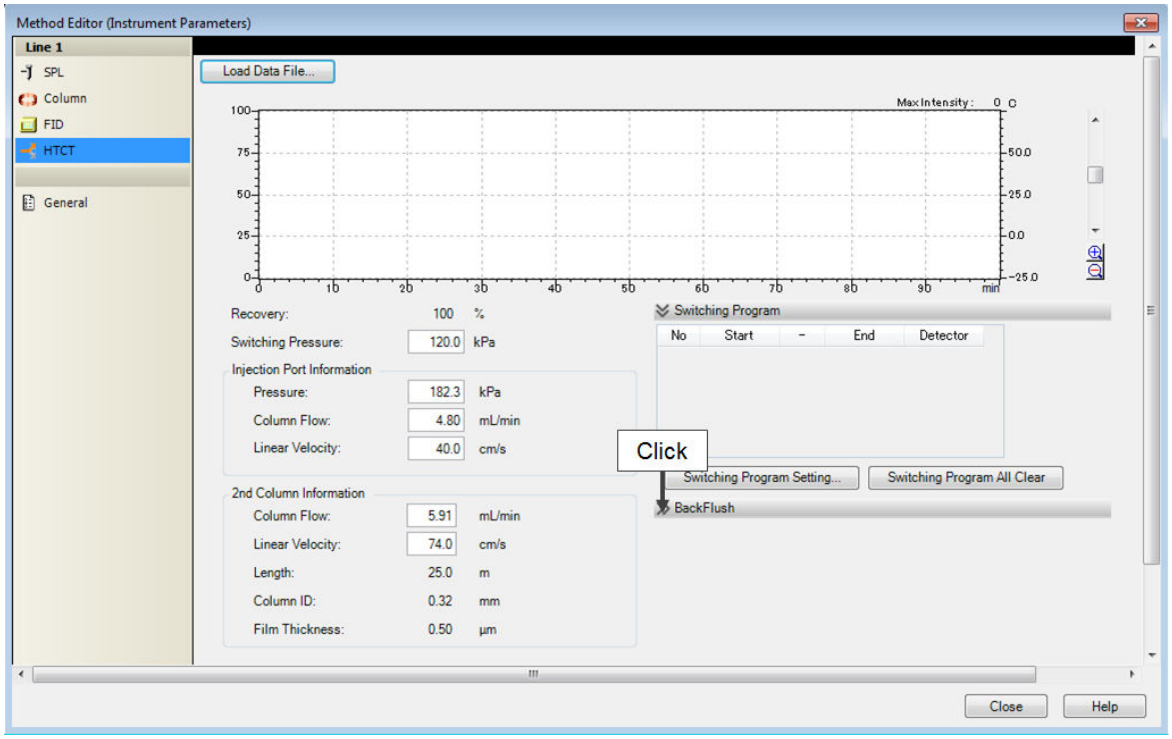
4 **Double-click the reference chromatogram display area to set [Back Flush Time].**



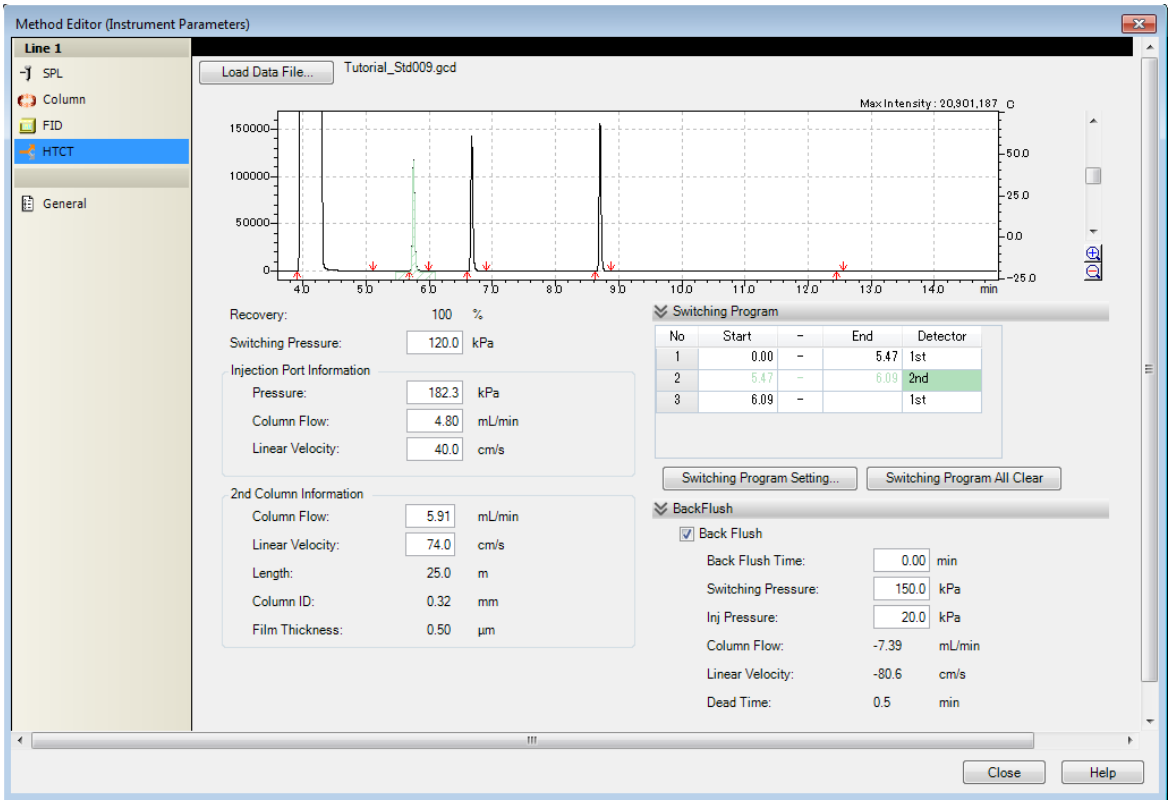
2.2.3 Back Flush by Using Other Types of AFT Systems

Even when AFT elements other than the back flush element are used, back flush is possible by controlling the APC pressure (switching pressure/splitting pressure) of each element. This section describes the back flush setting procedure when AFT elements other than the backflush element are used.

1 **Click the [Back Flush] group box.**



2 **Select the [Back Flush] checkbox, and set the back flush parameters.**



NOTE

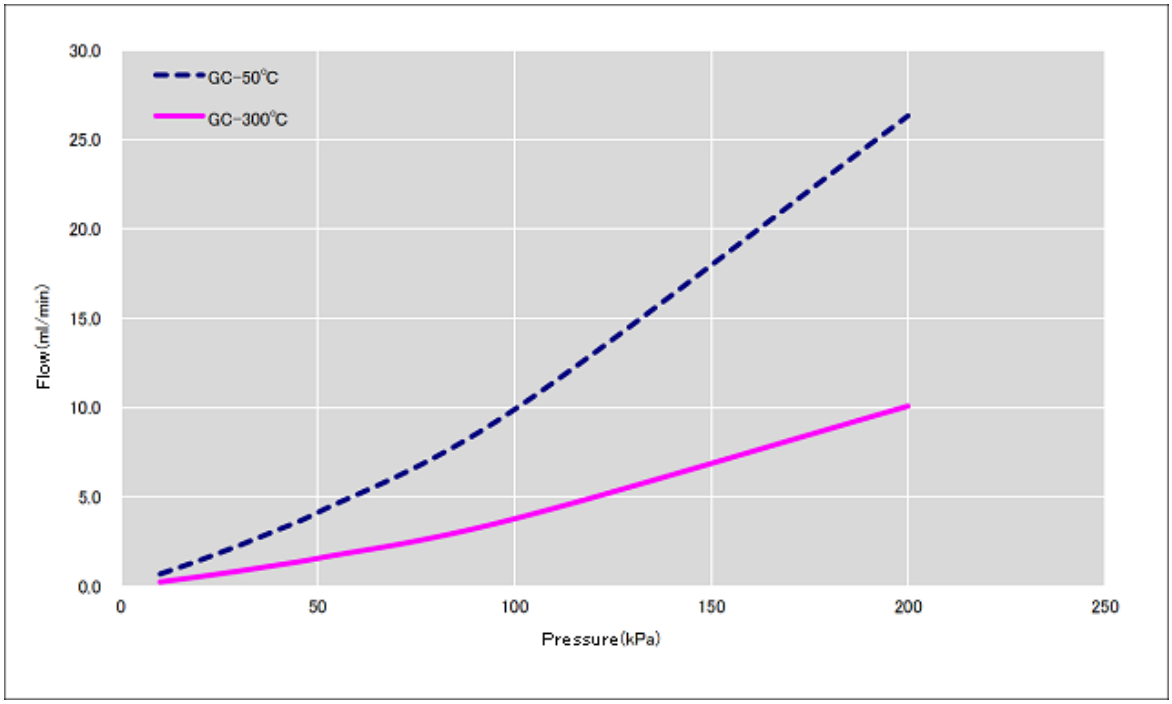
- It is not possible to configure the back flush time by double-clicking the reference chromatogram. Directly enter a value in [Back Flush Time].
- Be aware that setting the APC pressure too high may have an adverse impact, such as extinguishing the flame in the FID detector.

2.3 Warning Messages and Countermeasures

Message	Explanation	Countermeasure
Switching Pressure is higher than the injection port pressure.	This message is displayed when the APC pressure is higher than the injector pressure during analysis, because the carrier gas flow has reversed inside the column during analysis.	Lower the APC pressure. / Raise the injector pressure (pressure value in the sample injector window).
Injection port pressure is higher than switching Pressure.	This message is displayed when the injector pressure is higher than the APC pressure during back flush, because a back flush does not occur even when the back flush feature is actuated.	Raise the APC pressure / Lower the injector pressure.

Message	Explanation	Countermeasure
Excessive fluctuation of detector flow.	This message is displayed when the gas flowrate at the detector is 30 mL/min or higher, because there is a risk that the flame in the FID or other hydrogen flame detector will be extinguished, and the MS detector vacuum will be compromised.	Lower the APC pressure.
Column flow is larger than the total restrictor flow.	This is displayed when the volume of gas flowing through the column has become larger than the total volume of gas flowing through the restrictors, because part of the sample does not reach the detector.	Raise the APC pressure.

The following shows the correlation between the APC pressure and the restrictor carrier gas flowrate when the restrictor (0.15 mm × 0.5 m) provided as standard with the Shimadzu back flush element is used. Although it depends on the column oven temperature, it is recommended that you ordinarily set the APC pressure to 100 kPa or less during analysis.

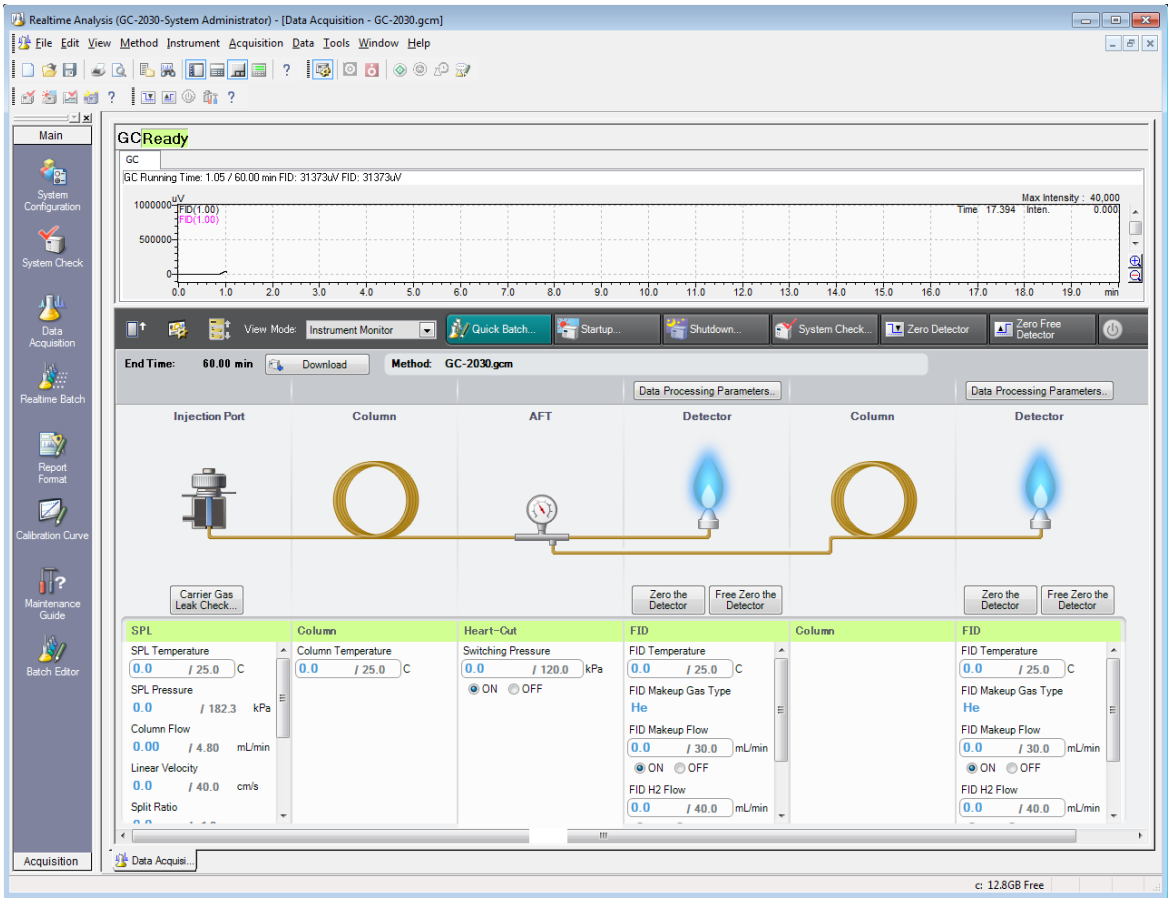


3 Heart-Cut

3.1 What Is the Heart-Cut Feature?

In a heart-cut system, components that cannot be separated with a single column are separated and loaded into the 2nd column by switching in a switching unit, and then detected by a second detector. A multidimensional gas chromatograph (MDGC) is another type of system with such a feature. A MDGC system typically uses two column ovens however, while the heart-cut system uses only one column oven.

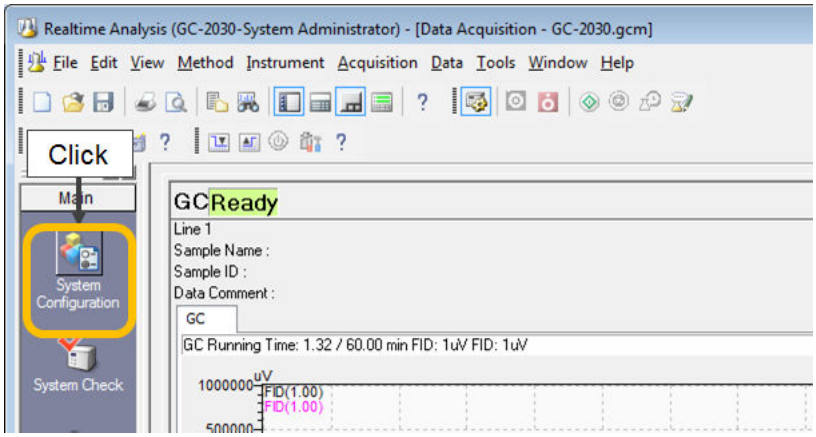
In a heart-cut system, multiple columns are connected and the flow lines are switched by control of the pressure supplied to the switching unit (switching pressure). Accordingly, the column linear velocity and flowrate are determined by the column inlet pressure and switching pressure unlike in ordinary analysis. Therefore, a special calculation method is required. In the LabSolutions [Method Editor (Instrument Parameters)] window, it is possible to configure and display the accurate column linear velocity and split ratio when the heart-cut system is actuated.



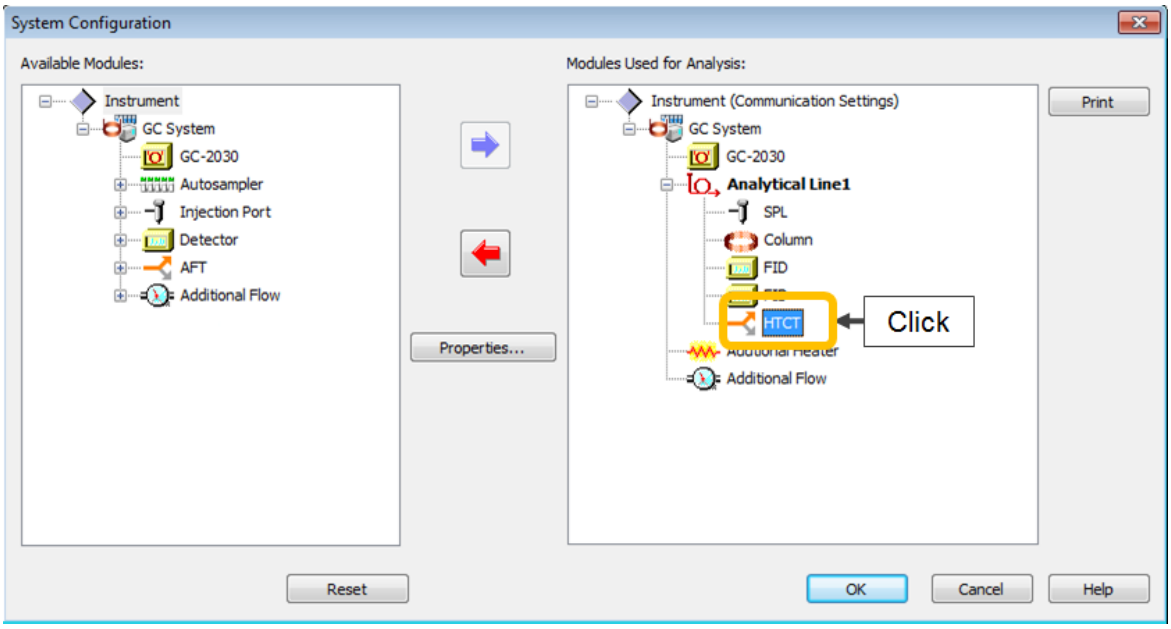
3.2 Configuring the 2nd Column Parameters

When performing a heart-cut analysis, the parameters for the 2nd column must be configured in the [System Configuration] window, using the following procedure.

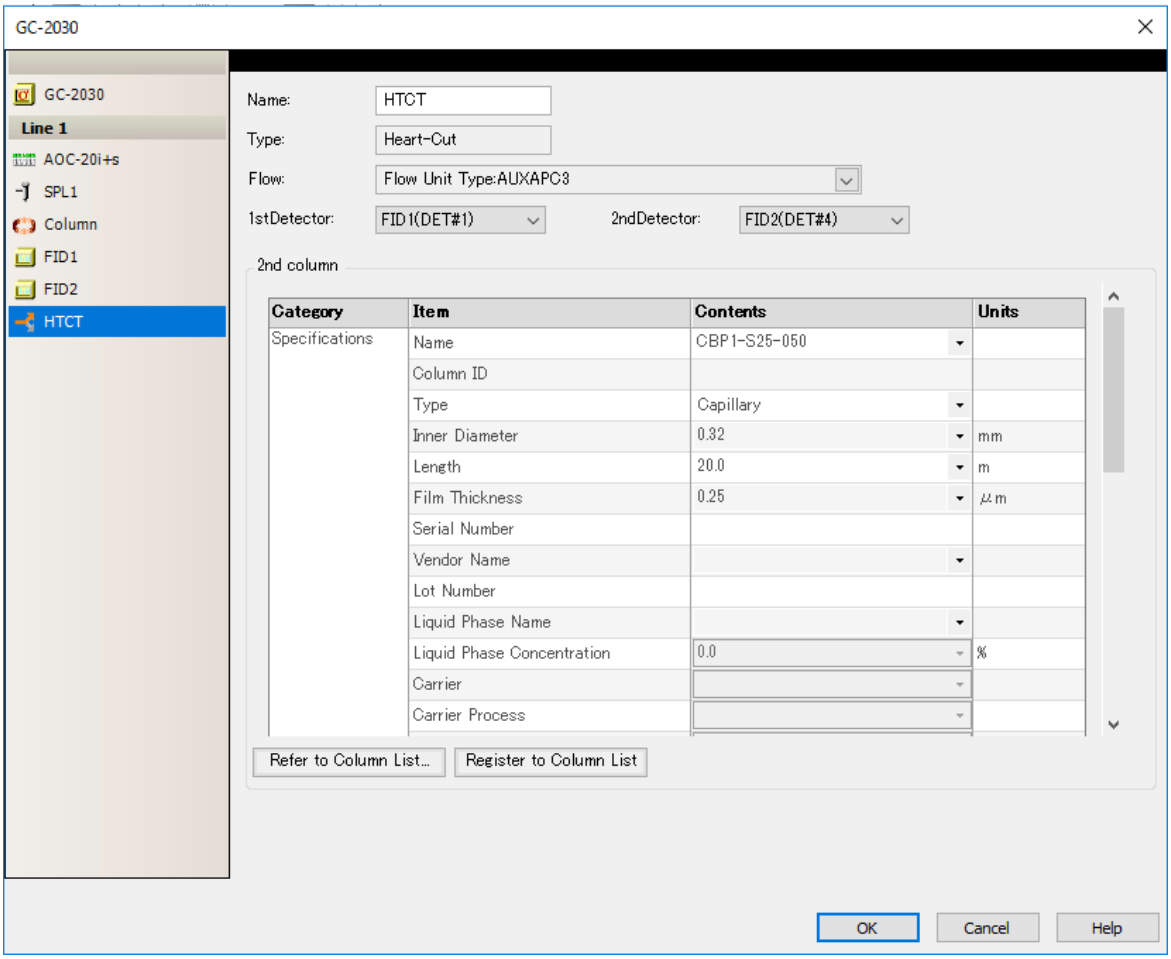
1 **Display the [System Configuration] window.**



2 **Click [(AFT name)] in the [System Configuration] window. (In this picture, click [HTCT].)**



3 Configure the parameters in the [2nd Column] area.

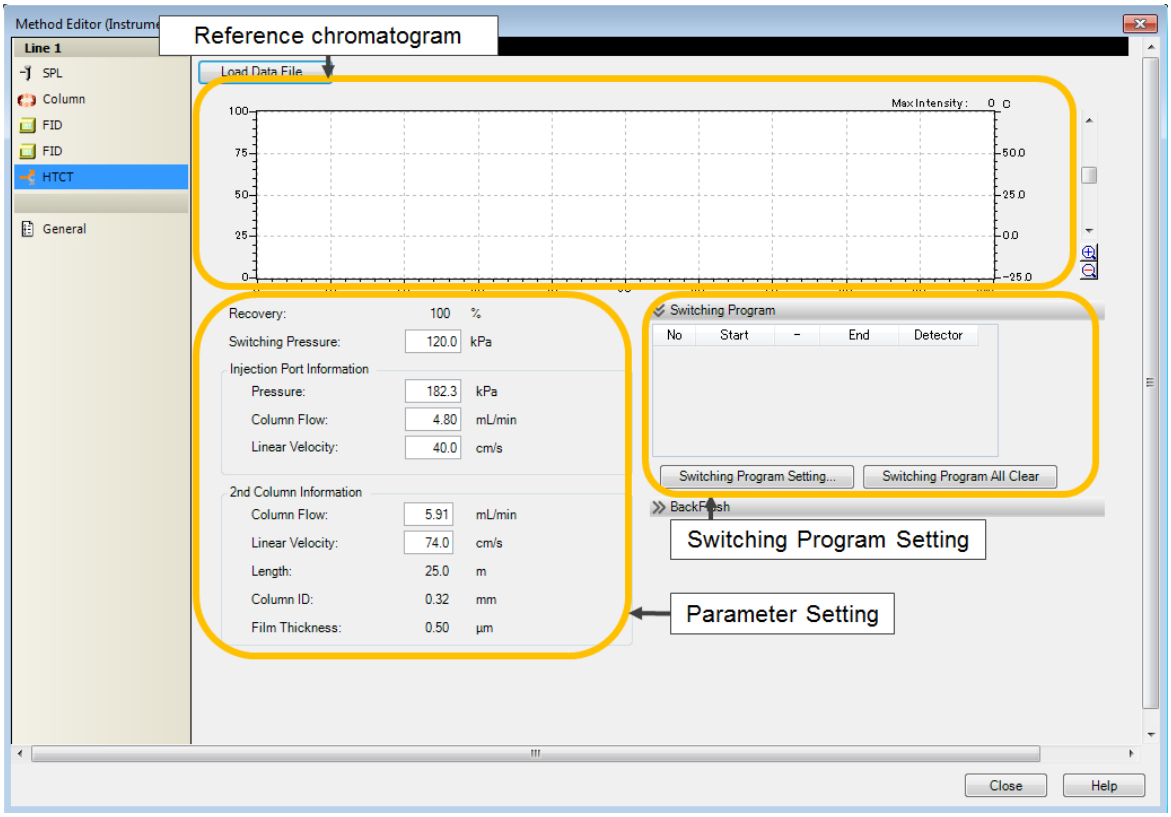


Hint By setting [1stDetector] and [2ndDetector], it is possible to correctly calculate gas flow rate when using a detector that is not at atmospheric pressure.

4 Click [OK] to apply the system configuration settings.

3.3 Editing an Instrument Method

3.3.1 [Method Editor (Instrument Parameters)] Window



Name	Explanation	Remarks
[Load Data File] button	Click this button to load data files to be used as a reference when configuring the switching program.	
[Recovery]	This indicates the switching recovery rate.	Reference "3.4 What Is the Switching Recovery Rate? "
[Switching Pressure]	Set the pressure to apply to the switching unit.	This corresponds to the 1st column's outlet pressure and the 2nd column's inlet pressure.
(Injection Port Information) Pressure	Set the pressure for the sample injector.	This corresponds to the 1st column's inlet pressure.
([2nd Column Information] area) [Column Flowrate] / [Linear Velocity]	Set the 2nd column's flowrate and linear velocity.	After these are configured, the optimal switching pressure is calculated and configured. This switching pressure is used for controlling the system.

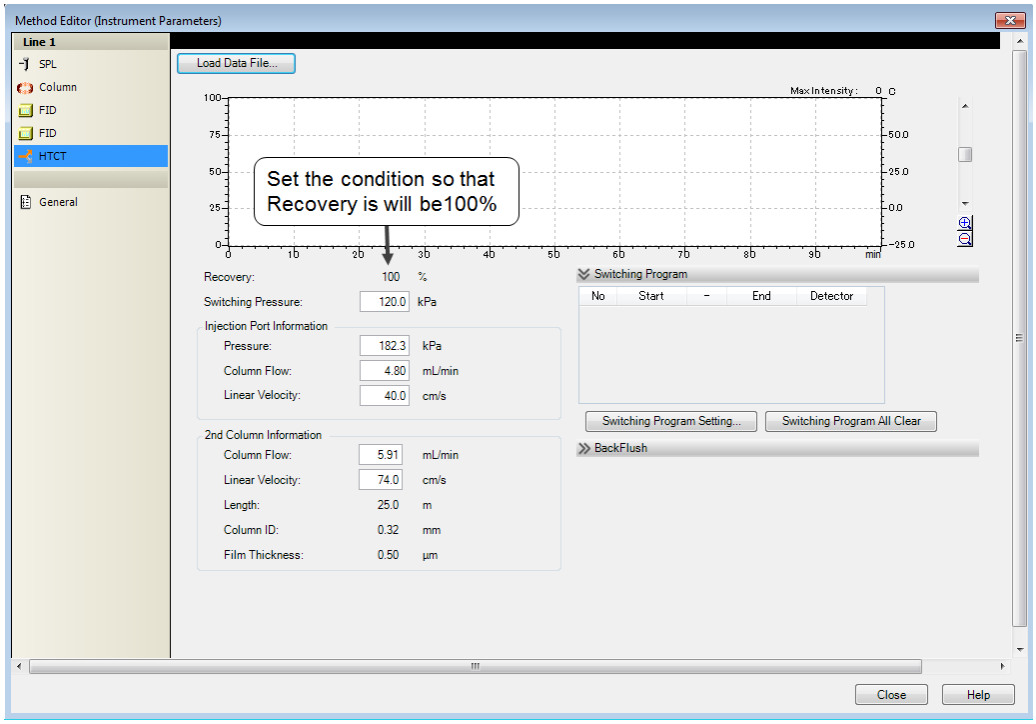
Name	Explanation	Remarks
[Switching Program]	Double-click the reference chromatogram to configure the switching program.	

Reference For the back flush settings, refer to"2.2.3 Back Flush by Using Other Types of AFT Systems"

3.3.2 Setting Procedure

1 **Perform an analysis using ordinary conditions, in which the heat-cut feature is not used.**

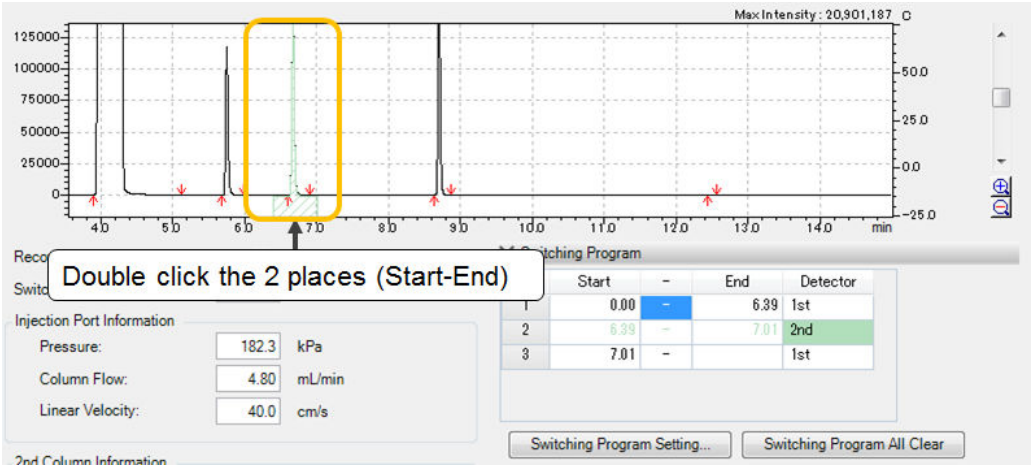
1. Configure the ordinary analysis conditions. This means that [Switching Program] is not Created.
2. Configure the switching pressure and the injector pressure so that the value in [Recovery] reaches "100" %. Then perform the analysis.



2 **Click [Load Data File] to load the file of data acquired from the analysis in step 1.**

3 **Create the switching program.**

1 In the reference chromatogram display area, double-click two time positions each to start and end the heart-cut feature.



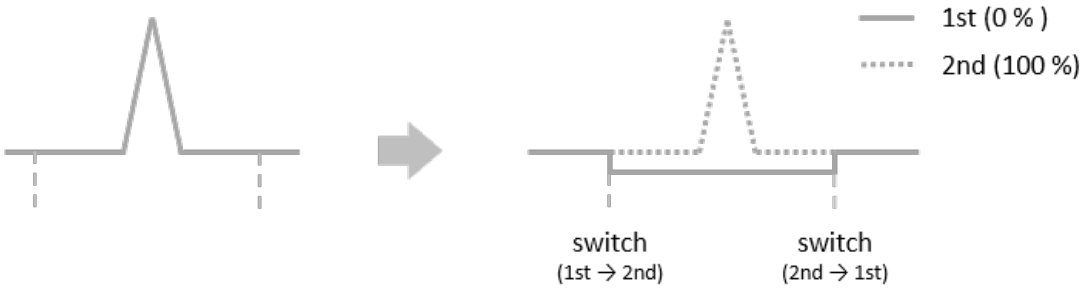
The switching program will be created. Components for peaks in the region shown with diagonal lines will be injected into the 2nd column for separation and detection during the next analysis.

Hint To delete or edit the switching program, display the window for editing the program by double-clicking the region shown with diagonal lines.

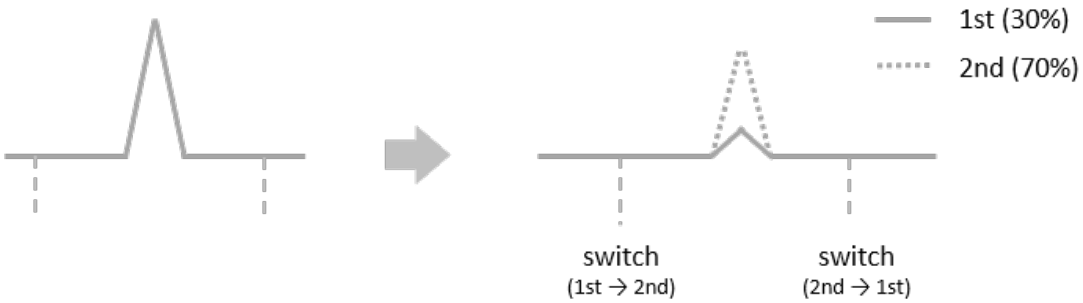
3.4 What Is the Switching Recovery Rate?

The switching recovery rate ([Recovery]) is an index that serves as a reference for what percentage of the target component moves from the 1st detector to the 2nd column.

Example: When the switching recovery rate is 100 %:



Example: When the switching recovery rate is 70 %:



Ordinarily, configure the settings so that the value in [Recovery] reaches "100" %. Note that the switching recovery rate varies significantly (several dozen %) depending on errors in the column dimensions. Only use it as a guide.

Hint

- The smaller the difference between the injector pressure and the switching pressure, the higher the recovery rate.
- If the difference between injector pressure and the switching pressure is constant, the larger the switching pressure, the higher the recovery rate.

3.5 Warning Messages and Countermeasures

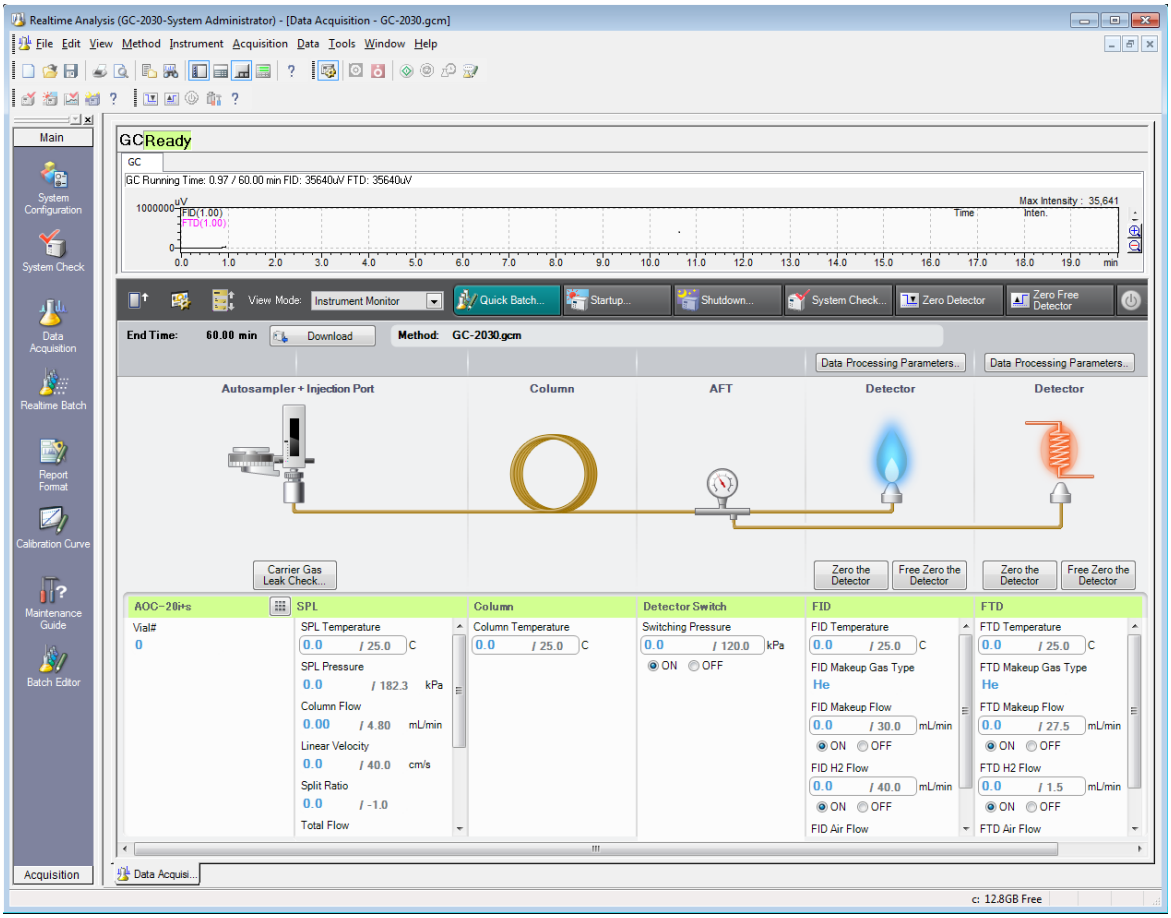
Message	Explanation	Countermeasure
Switching Pressure is higher than the injection port pressure.	This message is displayed when the switching pressure is higher than the sample injector pressure, because the carrier gas flow has reversed inside the column during analysis.	Lower the switching pressure. / Raise the injector pressure.

4 Detector Switching

4.1 What Is the Detector Switching Feature?

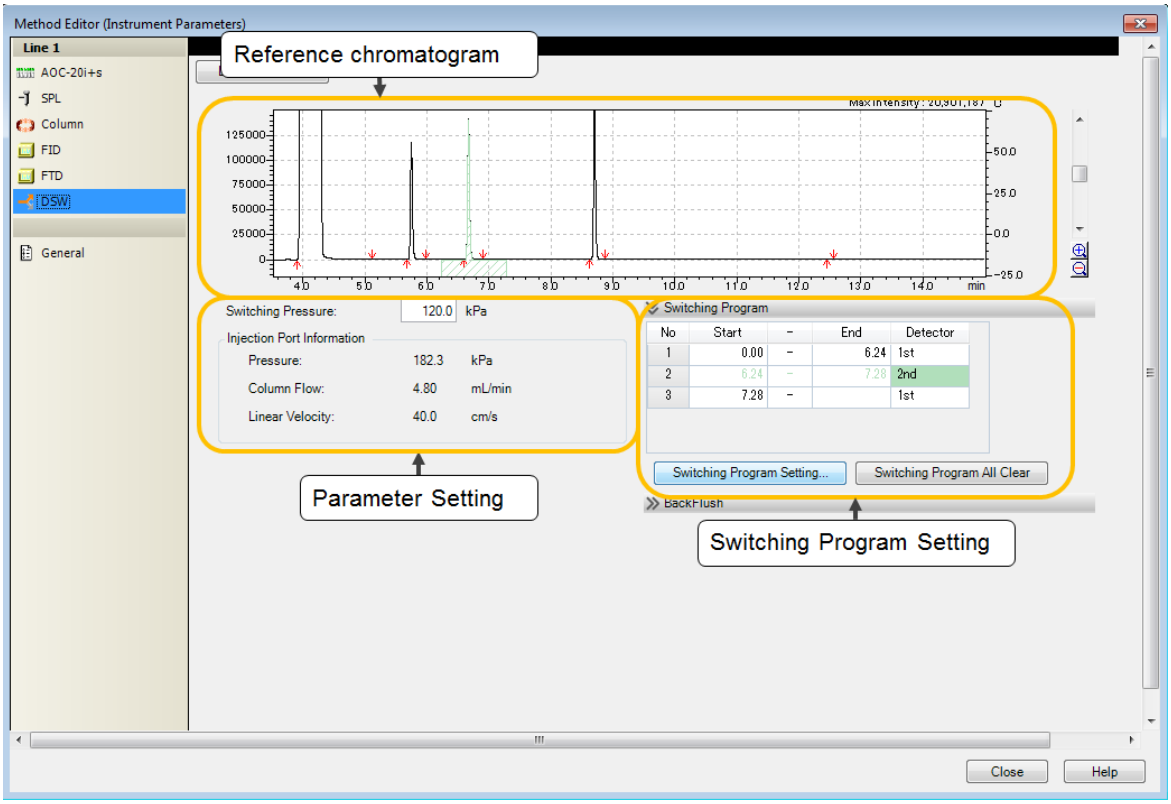
The detector switching system includes a switching unit at the column outlet, which enables samples to be divided to two detectors without column replacement. The system is also able to switch specified components only to a different detector during the same analysis.

Attaching the detector switching unit to the outlet of the analysis column makes it possible to divide the sample flow to two detectors without replacing the column. Multiple detectors are connected to the restrictors on the switching unit, and the flow lines are switched by controlling the pressure of the switching unit. Accordingly, unlike in ordinary analysis, the column linear velocity and flowrate are determined by the column inlet pressure and the pressure of the switching unit (hereinafter referred to as switching pressure), so a special calculation method is required. In the LabSolutions [Method Editor (Instrument Parameters)] window, it is possible to display the accurate column linear velocity and split ratio, as well as the ratio for splitting the sample flow to each detector, when the detector switching system is actuated.



4.2 Editing an Instrument Method

4.2.1 [Method Editor (Instrument Parameters)] Window

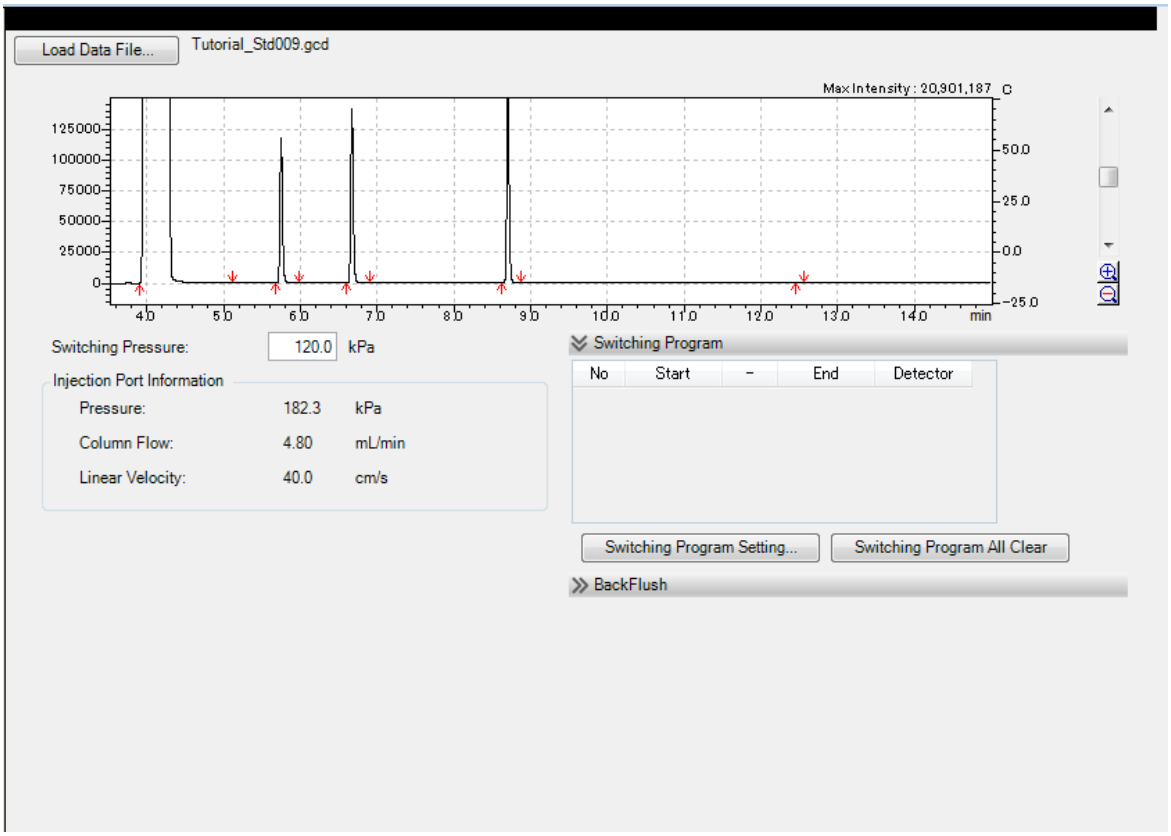


Name	Explanation	Remarks
[Load Data File] button	Click this button to load data files to be used as a reference when configuring the switching program.	
[Switching Pressure]	Set the pressure to apply to the switching unit.	
[Switching Program]	Double-click the reference chromatogram to configure the switching program.	

Reference For the details of the back flush settings, see "2.2.3 Back Flush by Using Other Types of AFT Systems".

4.2.2 Setting Procedure

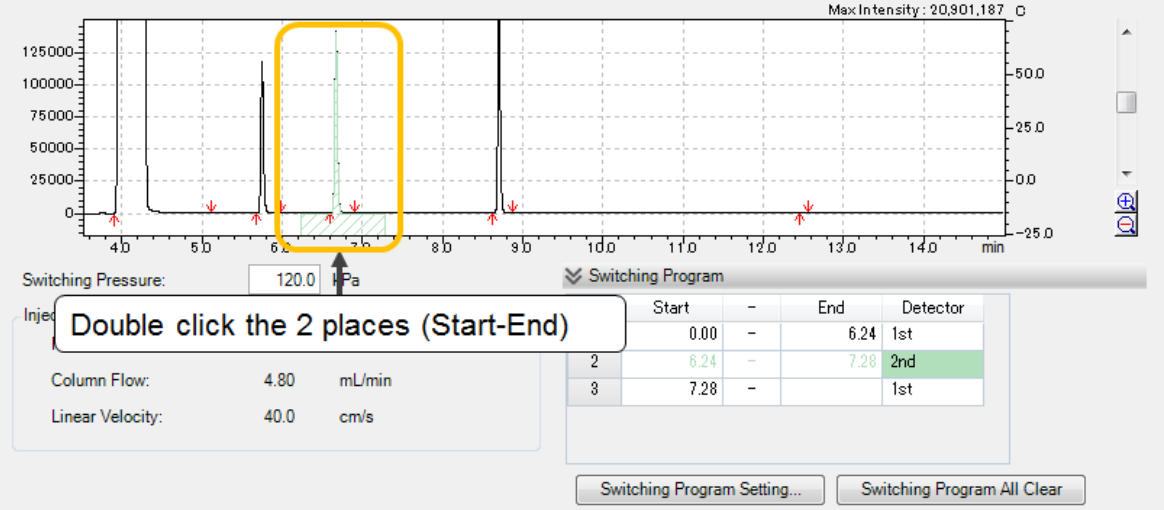
1 **Perform an analysis using ordinary conditions, in which the detector switching feature is not used.**



2 **Click [Load Data File] to load the file of data acquired from the analysis in step 1.**

3 **Create the switching program.**

In the reference chromatogram display area, double-click two time positions each to start and end the detector switching feature.



The switching program will be created. Components for peaks in the region shown with diagonal lines will be injected to the 2nd detector for detection during the next analysis.

Hint

- In order to always load the sample into the second detector throughout the analysis, click [Switching Program Setting] to display the [Switching Program Setting] window, and set the start time to 0.01 min and the end time to the analysis finishing time.
- To delete or edit the switching program, display the window for editing the program by double-clicking the region shown with diagonal lines.

4.3 Warning Messages and Countermeasures

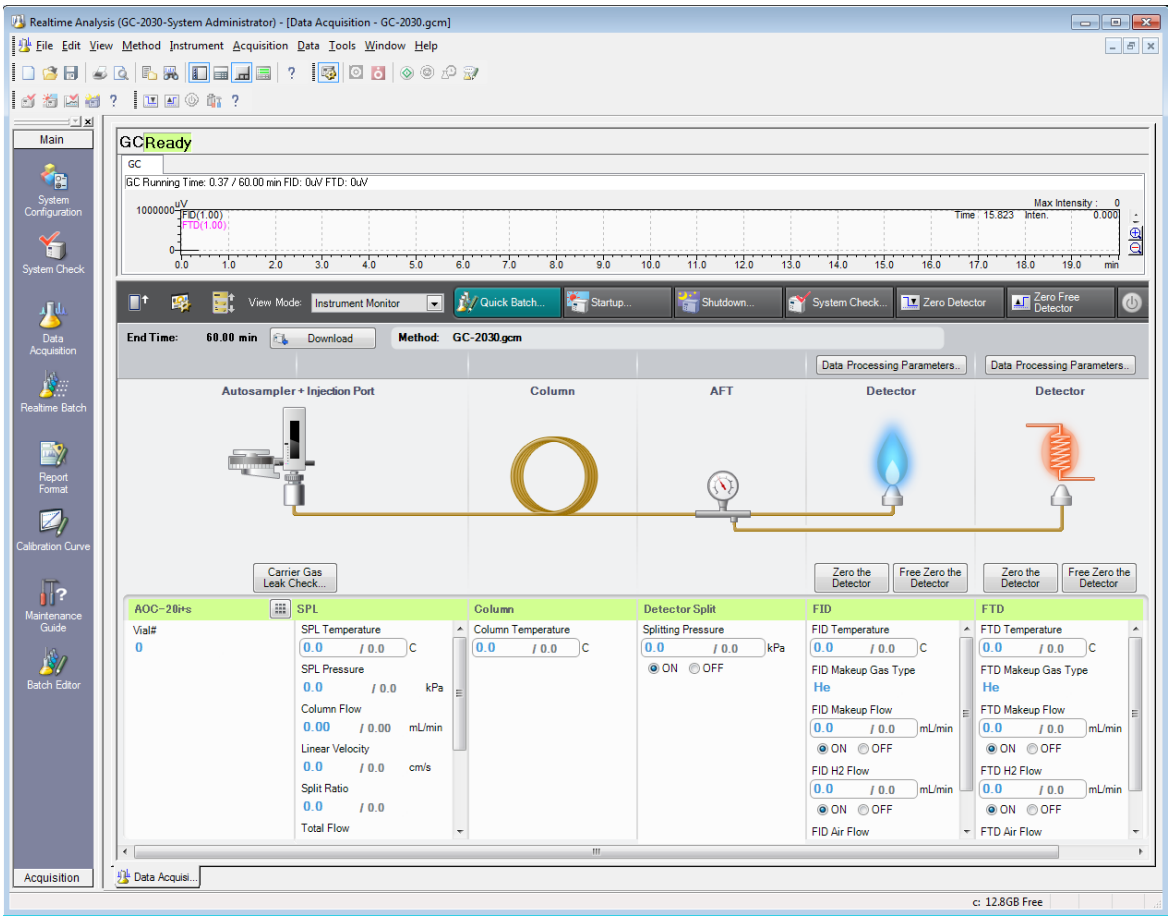
Message	Explanation	Countermeasure
Switching Pressure is higher than the injection port pressure.	This message is displayed when the switching pressure is higher than the sample injector pressure, because the carrier gas flow has reversed inside the column during analysis.	Lower the switching pressure. / Raise the injector pressure.

5 Detector Splitting

5.1 What Is the Detector Splitting Feature?

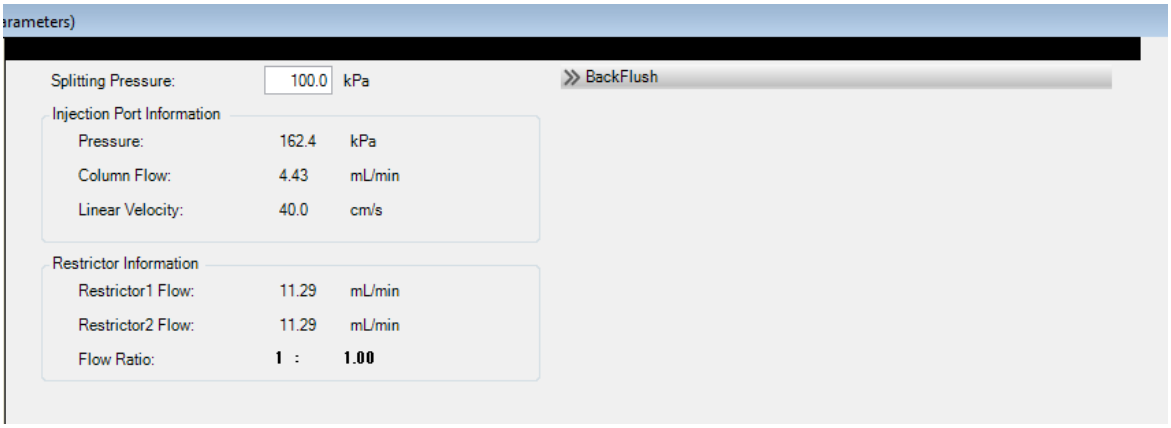
The detector splitting unit is used for splitting the column outlet so that sample components are detected by multiple detectors. Using this unit allow users to acquire a wealth of information from an analysis with a single sample injection.
Three types of the detector splitting unit is available, a 2-split unit that uses an APC (advanced pressure controller), a 3-split unit that does not use an APC, and a 2-split unit that does not use an APC.

Attaching the detector splitting element at the outlet of an ordinary analysis column enables a wealth of data to be obtained from a single analysis by loading the sample into multiple detectors.
Multiple detectors are connected to the restrictors on the splitting element. Accordingly, unlike in ordinary analysis, the column linear velocity and flowrate are determined by the column inlet pressure and the pressure of the splitting element (hereinafter referred to as the splitting pressure), so a special calculation method is required. In the LabSolutions [Method Editor (Instrument Parameters)] window, it is possible to set and display the accurate column linear velocity and split ratio, as well as the ratio for splitting the sample flow to each detector.



5.2 Editing an Instrument Method

5.2.1 [Method Editor (Instrument Parameters)] Window



Name	Explanation	Remarks
[Splitting Pressure]*	Set the pressure to apply to the splitting element.	Configure the pressure value to ensure that the flowrate for each restrictor does not exceed 20 mL/min.
[Restrictor Information]	Displays the split ratio and flowrate of the carrier gas flowing into each restrictor.	

*The splitting pressure is not displayed for systems in which an APC is not used at the splitting unit.

Reference For the details of the back flush settings, see "2.2.3 Back Flush by Using Other Types of AFT Systems".

5.3 Warning Messages and Countermeasures

Message	Explanation	Countermeasure
Switching Pressure is higher than the injection port pressure.	This message is displayed when the splitting pressure is higher than the injector pressure during analysis, because the carrier gas flow has reversed inside the column during analysis.	Lower the splitting pressure. / Raise the injector pressure (pressure in the sample injector window).

Message	Explanation	Countermeasure
Excessive fluctuation of detector flow.	This message is displayed when the gas flowrate at the detector is 30 mL/min or higher, because there is a risk that the flame in the FID or other hydrogen flame detector will be extinguished, and the MS detector vacuum will be compromised.	Lower the splitting pressure.
Column flow is larger than the total restrictor flow.	This is displayed when the volume of gas flowing through the column has become larger than the total volume of gas flowing through the restrictors, because part of the sample does not reach the detector.	Raise the splitting pressure.

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