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Oct. 2024

GC/MS Off-Flavor Analyzer 1.8.2

Instruction Manual 1.8

Read the instruction manual thoroughly before you use the product.

Keep this instruction 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 the installation, operation, usage cautions and accessories for this product. Read this manual thoroughly before using the product and operate the product in accordance with the instructions in this manual.

Also, keep this manual for future reference.

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⚠ WARNING	Indicates a potentially hazardous situation which, if not avoided, could result in serious injury or possibly death.
⚠ CAUTION	Indicates a potentially hazardous situation which, if not avoided, may result in minor to moderate injury or equipment damage.
NOTE	Emphasizes additional information that is provided to ensure the proper use of this product.
Hint	Provides useful information about operation of this system. Please read the description when required.
Reference	Indicates reference sections and pages.

⚠ WARNING

When the customer uses the DVD-ROM
This is a DVD-ROM disk. Do not play this on an audio DVD player, as the high volume may damage your hearing or the audio speakers.

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   3) Faults occurring after use in combination with hardware or software other than that designated by Shimadzu Corporation
   4) Faults in equipment and damage to data and software, including the OS, resulting from computer viruses
   5) Faults in equipment and damage to data and software, including the OS, resulting from power failures, including power interruptions and momentary voltage drops
   6) Faults in equipment and damage to data and software, including the OS, resulting from turning OFF the power switch on the equipment without following the proper shutdown procedures
   7) Faults not originating in the equipment itself
   8) Faults occurring after use in severe environmental conditions, such as those subject to high temperatures, high humidity levels, corrosive gases, or vibrations
   9) Faults resulting from fires, earthquakes and other natural disasters, contamination by radioactive or toxic substances, wars, riots, criminal activities, and other types of force majeure
   10) Faults occurring after the product is moved or transported following initial installation
   11) Faults occurring in consumable parts or parts dependent on them
   Note: Recording media, such as floppy disks and CD-ROMs, are also regarded as consumable parts.

- If there is a document such as a warranty provided with the product, or there is a separate contract agreed upon that includes warranty conditions, the provisions of those documents shall apply.
- Warranty periods for products with special specifications and systems are provided separately.

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1 About GC/MS Off-Flavor Analyzer

1.1 Overview

In recent years, there have been a string of food-related accidents and scandals. For example, there have been incidents of abnormal odor-causing components being detected in confections and pesticides being detected in frozen foods. In both cases, all the affected items were recalled from the market. In most incidents involving food, the problem was discovered when a consumer reported an abnormal odor. Therefore, to prevent incidents involving food, it is important to analyze the food for abnormal odors.

GC/MS systems, which are widely used for analyzing odors, allow you to investigate what components a sample contains (qualitative analysis), and how much of the components the sample contains (quantitative analysis). Analyzing odor components involves comparing data obtained by analyzing normal and abnormal samples to search for candidate components that might be causing the abnormal odor, then determining whether the sample contains the candidate components in concentrations higher than a threshold concentration. Components causing odors can also be identified by using a sniffer to actually confirm their smell.

However, analyzing odor components using a GC/MS system requires reviewing analytical conditions and performing data analysis, which can require a lot of work. It also requires obtaining sensory information and threshold values for the odor components, which can be a difficult task for analysts with limited knowledge or experience.

In contrast, the GC/MS odor analysis system already includes a database (Smart Database) of parameters, sensory information (such as types of odors and odor threshold values), and other data for about 150 of the primary compounds that cause odors. That means even less experienced or knowledgeable analysts can use the system to immediately start analyzing odor components.

1.8.2

Retention indices calculated from n-alkanes using columns with three types of liquid phases are registered in the Smart Database. Consequently, by simply analyzing a standard n-alkane mixture sample and using the automatic adjustment of retention time (AART) function, the detected retention times can be easily predicted for registered odor components. Furthermore, the Smart MRM function can be used to automatically create analytical methods from the database. Calibration curves for semi-quantitative analysis are also registered in created methods, so that semi-quantitative values can be calculated for detected components.

1.8

1.8.1

1.8.2

The GC/MS odor analysis system also supports using headspace, SPME, and various other pretreatment processes or a sniffer. That allows you to select the pretreatment process

best suited for a given sample.

This manual has been created on the assumption that the reader already has basic knowledge on the operation of Shimadzu's GC-MS(/MS) and GCMSsolution. Accordingly, names and terms specific to GCMSsolution are used throughout this manual. It is recommended that first-time users and those unsure of any points refer to the instruction manual for GC-MS(/MS), GC/MS Operation Guide, and GCMSsolution Help. For details on how to use the various pretreatment units, refer to the instruction manual for the applicable pretreatment unit.

NOTE

This manual has been created on the assumption that the reader already has basic knowledge on the operation of Shimadzu's GC-MS(/MS) and GCMSsolution. Accordingly, names and terms specific to GCMSsolution are used throughout this manual. It is recommended that first-time users and those unsure of any points refer to the instruction manual for GC-MS(/MS), GC/MS Operation Guide, and GCMSsolution Help. For details on how to use the various pretreatment units, refer to the instruction manual for the applicable pretreatment unit.

1.2 Operating Environment

The GC/MS Off-Flavor Analyzer requires the following software environment.

OS	Microsoft Windows 7 Professional, Microsoft Windows 10 Professional Microsoft Windows 11 Professional
Excel	Microsoft Excel 2016 (32-bit version), Excel 2019 (32-bit/64-bit version), Excel 2021 (32-bit/64-bit version)
Workstation software	GCMSsolution Ver. 4.31 or later (When using Excel 64-bit version, Ver. 4.53 or later is required.) Evolution Workstation Ver. 4.6.3 (for OPTIC-4 control)
Firmware	OPTIC-4 Firmware Ver. 4.5.11

1.3 Instruments Supported

The GC/MS Off-Flavor Analyzer can be used with the following instruments.

1.8.2 GCMS-TQ series: GCMS-TQ8040NX/TQ8050NX/TQ8040/TQ8050/TQ8030

GCMS-QP series: GCMS-QP2020NX/QP2020/QP2010 Ultra

The following consumables are recommended.

Columns

Low-polarity column

GL Sciences Inc. InertCap 5MS/Sil
(30 m x 0.32 mm I.D., df = 0.5 µm)
P/N: 1010-15244

Mid-polarity column

GL Sciences Inc. InertCap 17MS
(30 m x 0.25 mm I.D., df = 0.25 µm)
P/N: 1010-20142

High-polarity column

GL Sciences Inc. InertCap Pure-Wax
(30 m x 0.25 mm I.D., df = 0.25 µm)
P/N: 1010-68142

Insert liner

For loading samples by the liquid injection method or the headspace method:

Shimadzu Corporation Capillary glass insert (split/splitless, 95 mm, deactivated, with wool) P/N:227-35007-01

For loading samples by the solid phase micro extraction method (SPME method):

Shimadzu Corporation SPME Liner P/N:092089

For loading samples by the solid phase micro extraction method (SPME Arrow method):

Shimadzu Corporation SPME Arrow Liner P/N:227-35390-03

SPME Fibers

For loading samples manually:

SUPELCO 50/30µm DVB/CAR/PDMS P/N:57328-U

For loading samples by an autosampler:

Shimadzu Corporation 50/30µm DVB/CAR/PDMS P/N:227-35345-01

SPME Arrow Fibers

For loading samples by an autosampler:

Shimadzu Corporation 120µm DVB/CAR/PDMS P/N:227-35336-01

1.4 GC/MS Off-Flavor Analysis Composition

1.4.1 Contents

This GC/MS Off-Flavor Analyzer includes a DVD-ROM, Instruction Manual and Handbook. If this is insufficient, kindly contact Shimadzu Corporation.

P/N	Item	Quantity
225-30394-91	DVD-ROM	1
225-28439	GC/MS Off-flavor Analyzer Instruction Manual	1
225-28455	GC/MS Off-flavor Analyzer Handbook	1
225-28470	GC/MS Off-flavor Analyzer QuickGuide	1

The DVD-ROM includes the Smart Database installer and the PDF version of the User's Guide.

NOTE

This product does not include columns. Be sure to obtain the specified ones.

1.4.2 Details of Files Installed

Execution the GC/MS Off-Flavor Analyzer installer (Setup.exe) installs the following files.

Note: "C:" refers to the drive where GCMSsolution is actually installed.

a) Method Files and Batch Files

Files for columns are installed in the following folders corresponding to respective columns.

For InertCap 5MS/Sil :

"C: \GCMSsolution\SmartDatabase\Off-flavor\InertCap_5MS-Sil"

For InertCap 17MS :

"C: \GCMSsolution\SmartDatabase\Off-flavor\InertCap_17MS"

For InertCap_Pure-Wax :

"C: \GCMSsolution\SmartDatabase\Off-flavor\InertCap_Pure-Wax"

File Name	Description
Method Files	
TQ_MS_(Column Name)_AART.qgm SQ_MS_(Column Name)_AART.qgm TQ_Sniff_(Column Name)_AART.qgm SQ_Sniff_(Column Name)_AART.qgm	Method file for adjusting retention times *1

File Name	Description
Method Files	
TQ_MS_(Column Name)_Template.qgm SQ_MS_(Column Name)_Template.qgm TQ_Sniff_(Column Name)_Template.qgm SQ_Sniff_(Column Name)_Template.qgm	Template file for creating method file from the database *1
TQ_MS_(Column Name)_Correct_SIM.qgm TQ_MS_(Column Name)_Correct_MRM.qgm SQ_MS_(Column Name)_Correct.qgm TQ_Sniffer_(Column Name)_Correct_SIM.qgm TQ_Sniffer_(Column Name)_Correct_MRM.qgm SQ_Sniffer_(Column Name)_Correct.qgm	Method files for analyzing calibration curve correction *1, *2
TQ_MS_(Column Name)_QC.qgm SQ_MS_(Column Name)_QC.qgm TQ_Sniffer_(Column Name)_QC.qgm SQ_Sniffer_(Column Name)_QC.qgm	Method files for analyzing accuracy control *1
Batch Files	
TQ_MS_(Column Name)_QC_SIM.qgb TQ_MS_(Column Name)_QC_MRM.qgb SQ_MS_(Column Name)_QC.qgb TQ_Sniff_(Column Name)_QC_SIM.qgb TQ_Sniff_(Column Name)_QC_MRM.qgb SQ_Sniff_(Column Name)_QC.qgb	Batch files for adjusting retention time, calibration curve correction, and accuracy control analysis *2

*1 One of the four files on the left is installed, as appropriate, depending on the type of instrument selected during installation and whether or not a sniffer is included.

*2 Because MRM and SIM functions can be used in TQ series systems, batch files and method files for calibration curve correction are provided for MRM and SIM modes.

b) Parameter Files for Pretreatment Unit

Cycle files for AOC-5000 Plus units are installed in the "C:\ProgramData\Shimadzu\XML" folder.

File Name	Description
Off-flavor_Liquid.XML	Liquid injection cycle file
Off-flavor_HS.XML	HS cycle file
Off-flavor_SPME.XML	SPME cycle file

c) Report Files

Report files are installed int the "C:¥GCMSsolution¥SmartDatabase¥Off-flavor" folder.

File Name	Description
Off-flavor_QC.qgr	Report file for accuracy control analytical results
Off-flavor_Report.qgr	Report file various analytical results

d) Smart Database Files

Smart Database files are installed in the “C:¥GCMSsolution¥SmartDatabase” folder.

File Name	Description
Off-flavor_SQ_MS_5MS.xlsm Off-flavor_TQ_MS_5MS.xlsm Off-flavor_SQ_Sniff_5MS.xlsm Off-flavor_TQ_Sniff_5MS.xlsm	Smart Database files for InertCap 5MS/Sil *1
Off-flavor_SQ_MS_17MS.xlsm Off-flavor_TQ_MS_17MS.xlsm Off-flavor_SQ_Sniff_17MS.xlsm Off-flavor_TQ_Sniff_17MS.xlsm	Smart Database files for InertCap 17MS *1
Off-flavor_SQ_MS_Wax.xlsm Off-flavor_TQ_MS_Wax.xlsm Off-flavor_SQ_Sniff_Wax.xlsm Off-flavor_TQ_Sniff_Wax.xlsm	Smart Database files for InertCap_Pure-Wax *1

*1 One of the four files on the left is installed, ad appropriate, depending on the instrument type (SQ or TQ) selected during installation and whether or not a sniffer is included.

e) Library Files

Library Files are installed in the “C: \GCMSsolution\Library”.

File Name	Description
OFF-FLAVOR_GCMS.LIB	Mass spectral library file
OFF-FLAVOR_SNIFFING.LIB	Mass spectral library file (if a sniffer is used)

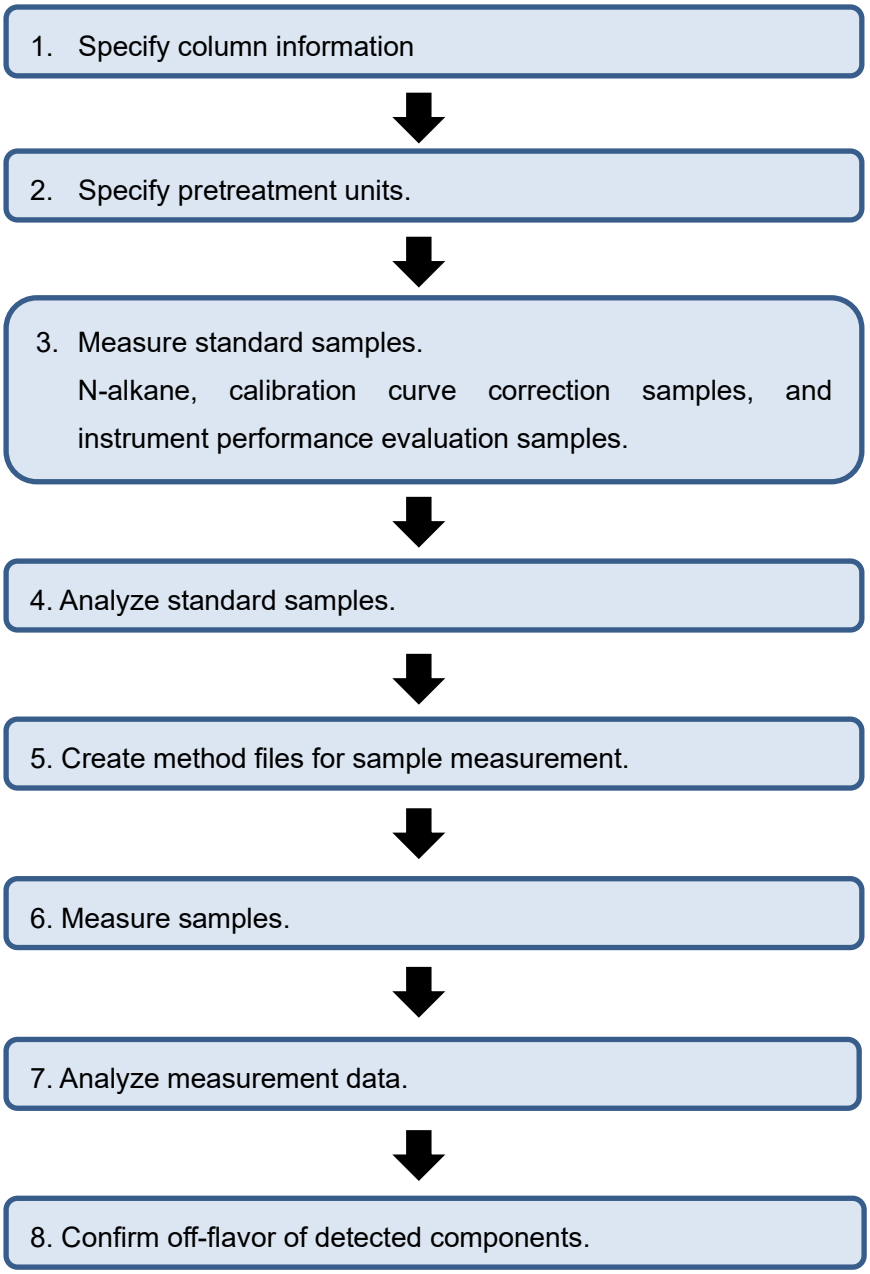
f) Manual

Manual file is installed in the “C: \GCMSsolution\Manual\Off-flavor”.

File Name	Description
Off-flavor_ Analyzer_ Manual.pdf	Manual (This one)

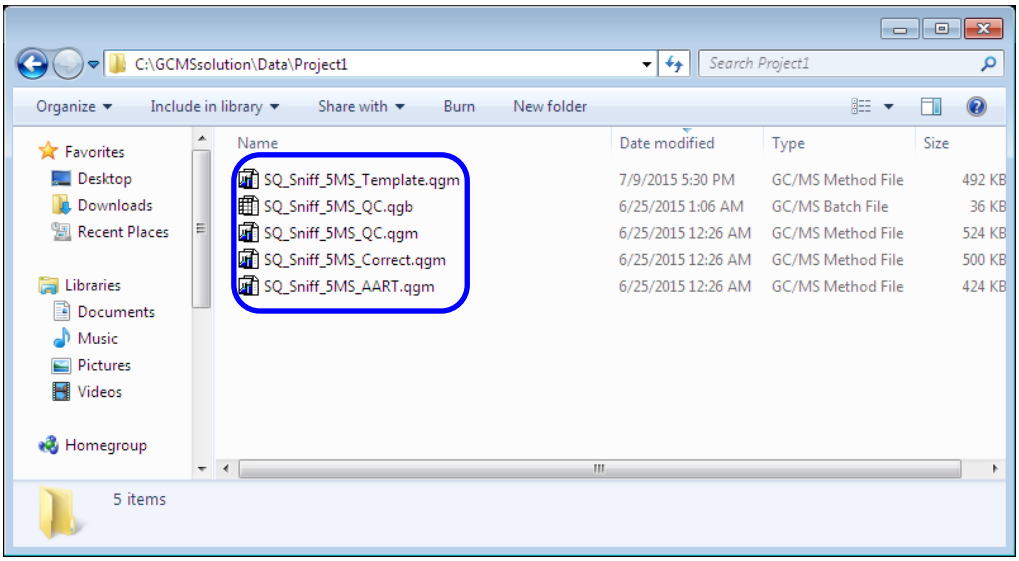
2 Usage Procedures

Method files are created and off-flavor analyzed according to the following process flow.



2.1 Preparation

1) Create a folder for saving measurement method and data files.
2) Copy all files from the "C:\GCMSsolution\SmartDatabase\Off-flavor\(column name)" folder and paste them in the folder that was created. Specify either "InertCap_5MS-Sil," "InertCap_17MS," or "InertCap_Pure-Wax" as the column name. Select the folder corresponding to the column used.

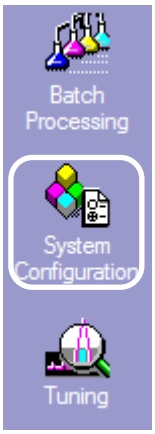


2.2 Specifying Column Information

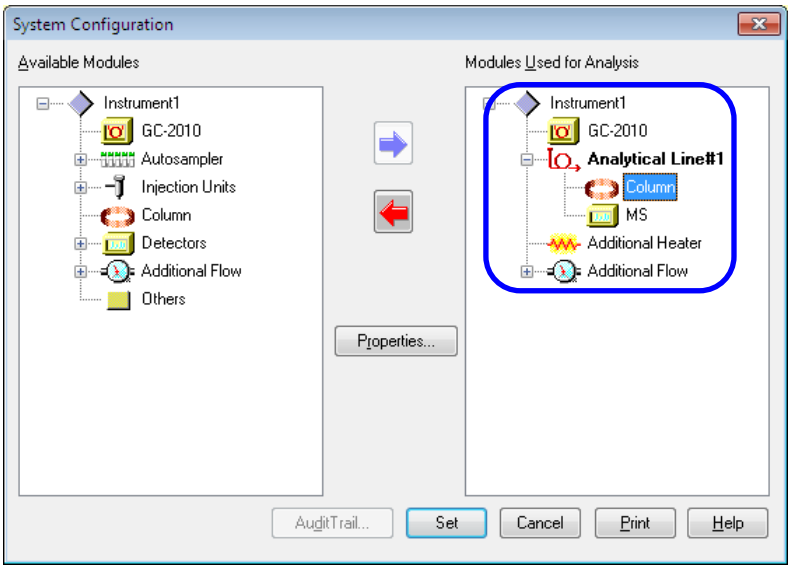
Register information about the column used in GC/MS Off-Flavor Anaylzer, as follows.

1) Run the [GCMS Real Time Analysis] program.

2) Click the [System Configuration] icon on the assistant bar.

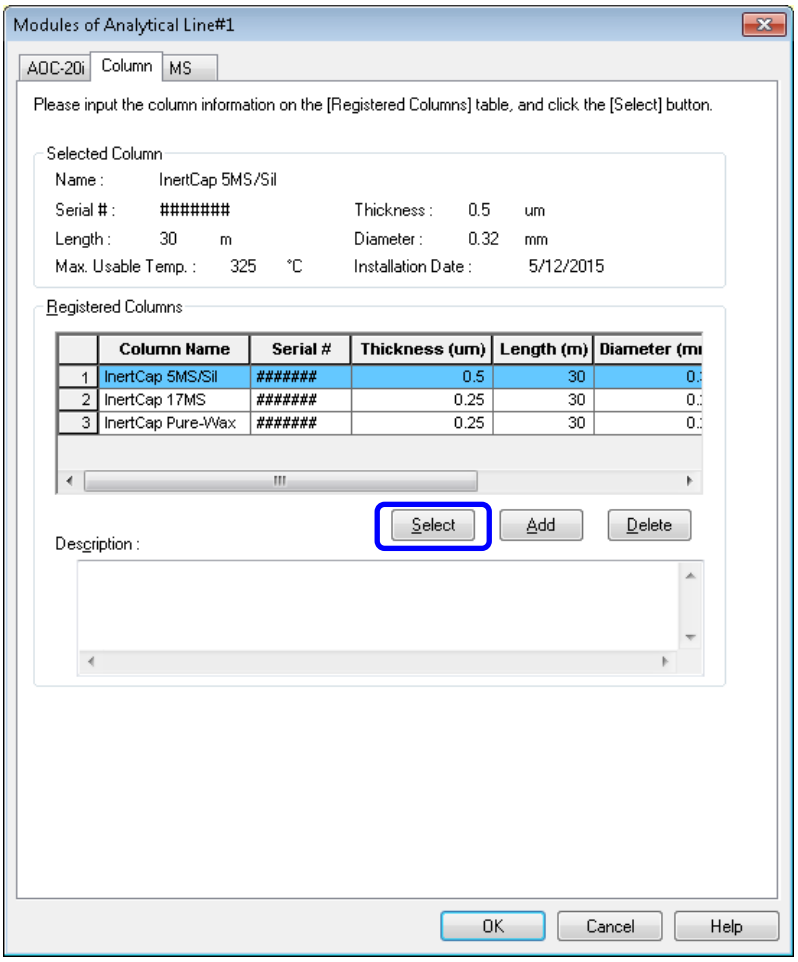


3) Add the GC unit and column to the [Modules Used for Analysis] list.



4) Select the column and click [Properties].

5) Click [Add] to register additional column information. Enter the following information for the column to be used.



> **Hint**
>
> The detection limit for target components differs depending on the polarity of the column used. For information about the relationship between column polarity and the detection limit, refer to "*Appendix 4. Registered Compound Information in the GC/MS Off-Flavor Analyzer Handbook*.".

> **NOTE**
>
> When a retention index registered in a database is used, be sure to use a column from the above-mentioned manufacturer matching the specified liquid phase type, length, internal diameter, and thickness.

6) Select the colun currently installed in the GC/MS system and then click [Select].

7) Click [OK].

2.3 Specifying Pretreatment Units

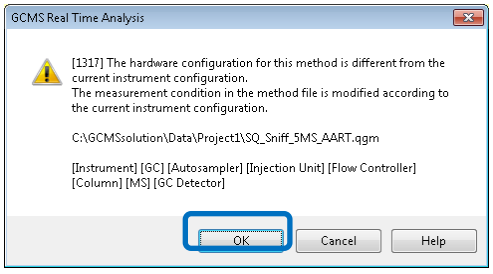
Specify system configuration settings based on the pretreatment unit used. Also specify pretreatment parameter settings in the measurement method file. If a pretreatment unit is used, specify the settings as described in "*2.3.1 Using an AOC-20i Autoinjector*" if using an AOC-20i or AOC-30i unit, in "*2.3.2 Using an AOC-5000 Plus Autoinjector*" if using an AOC-5000 Plus or AOC-6000 *series*, or in "*2.3.3 Using an OPTIC-4 Inlet*" if using an OPTIC-4 unit, or in "*2.3.4 Using an TD-30 series*" if using an TD-30 series.

NOTE

For the remaining procedure steps, the method, batch, Smart Database file names used for GCMS-QP series systems are indicated. If using a GCMS-TQ series system, read "SQ" as "TQ" wherever it appears in file names.
Example: Read "SQ_Sniff_(column name)_AART.qgm" as "TQ_Sniff_(column name)_AART.qgm."

NOTE

If the message "[1317] The hardware configuration for this method is different from the current instrument configuration. The measurement condition in the method file is according to the current instrument configuration." is displayed when the method file for adjusting retention time is loaded in the [GCMS Real Time Analysis]-[Acquisition] window during the operations described below, click [OK].



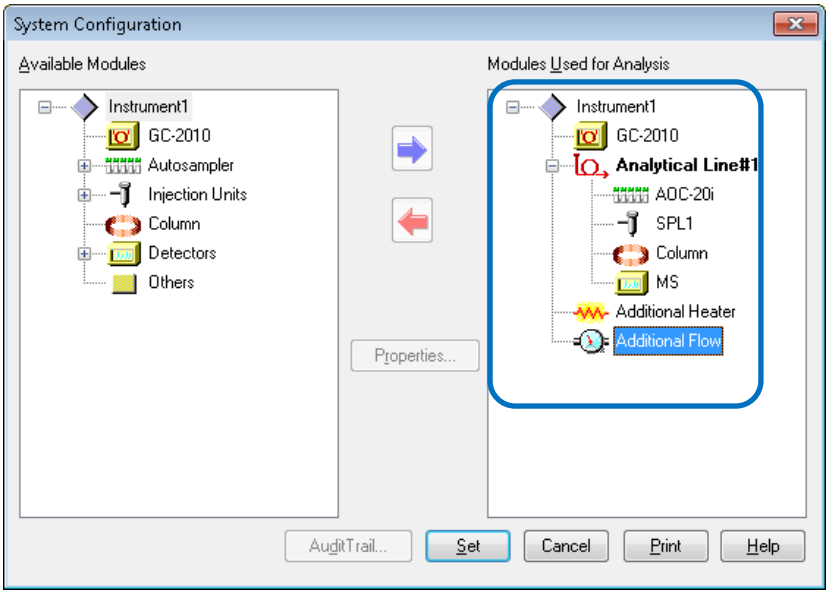
NOTE

If the OPTIC-4 is installed onto the instrument, do not turn OFF the OPTIC-4 even when it is not being used in the system. In addition, ensure that the Evolution Workstation is always running.

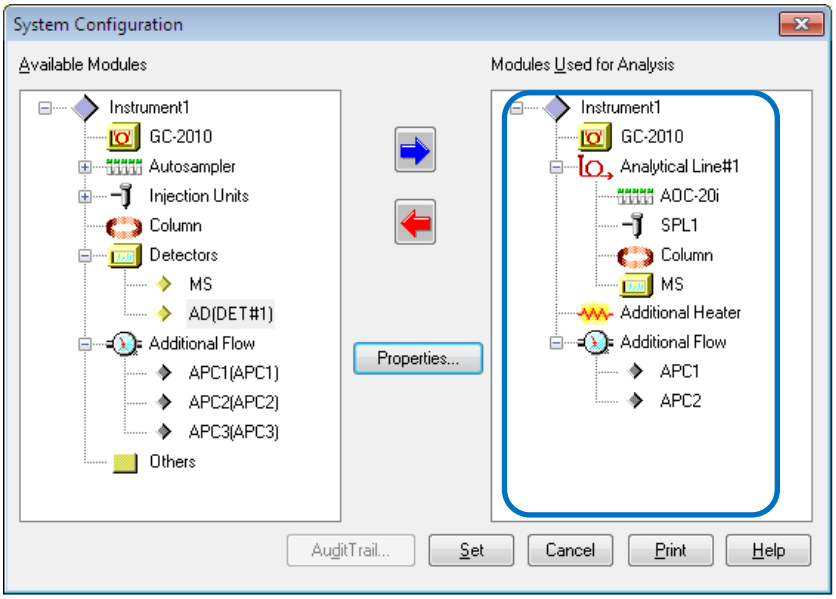
2.3.1 Using an AOC-20i/AOC-30i Autoinjector

1) Specify modules in the [Modules Used for Analysis] list as shown below.

If a Sniffer is Not Used



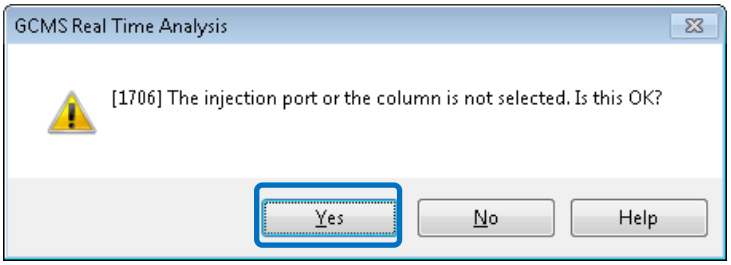
If a Sniffer is Used



2) Click the [Set] to complete the environment configuration

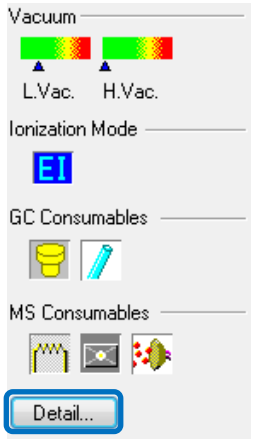
NOTE

If a sniffer is used and the "[1706] The injection port or the column is not selected. Is this OK?" message is displayed, click [Yes].

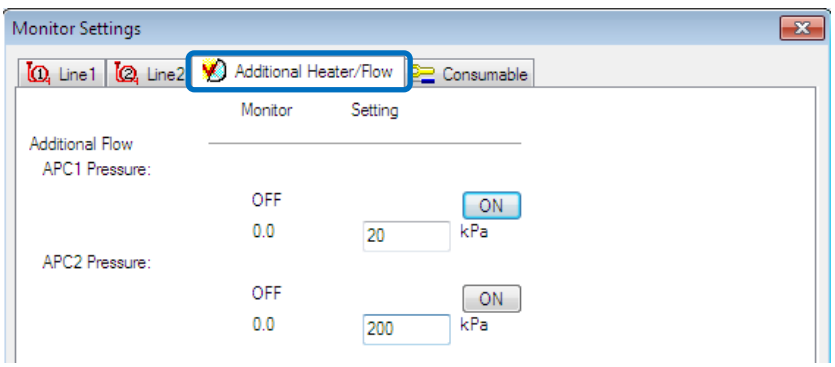


If a sniffer is used, switch the APC pressure ON, as described in steps 3) to 5). If a sniffer is not used, skip to step 6).

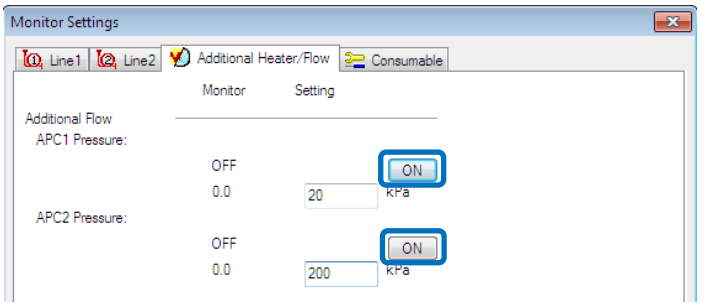
3) Click [Detail].



4) Click the [Additional Heater/Additional Flow] tab.



5) Click the ON/OFF button to switch [APC1] and [APC2] to [ON].



6) In the [Acquisition] window, open and overwrite the following method files, located in the folder created in "*2.1 Preparation*", to ensure configuration settings are consistent with the instruments that you use.

Method files for adjusting retention times

"SQ_MS_(column name)_AART.qgm" or "SQ_Sniff_(column name)_AART.qgm"

Method file for analyzing accuracy control

"SQ_MS_(column name)_QC.qgm" or "SQ_Sniff_(column name)_QC.qgm"

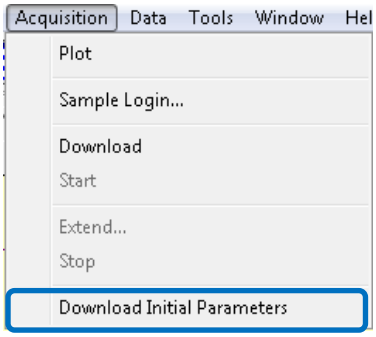
Method file for correcting calibration curve

"SQ_MS_(column name)_Correct.qgm" or "SQ_Sniff_(column name)_Correct.qgm"

Method file for Template

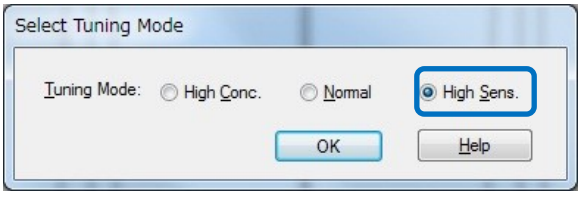
"SQ_MS_(column name)_Template.qgm" or "SQ_Sniff_(column name)_Template.qgm"

7) Click [Download Initial Parameters] on the [Acquisition] menu with the last method file loaded. Parameter settings are sent to the instrument.



8) Wait until each parameter setting changes to the specified settings and the [GC] and [MS] settings change to [Ready].

9) Perform auto-tuning, using the [High Sens.] tuning mode.

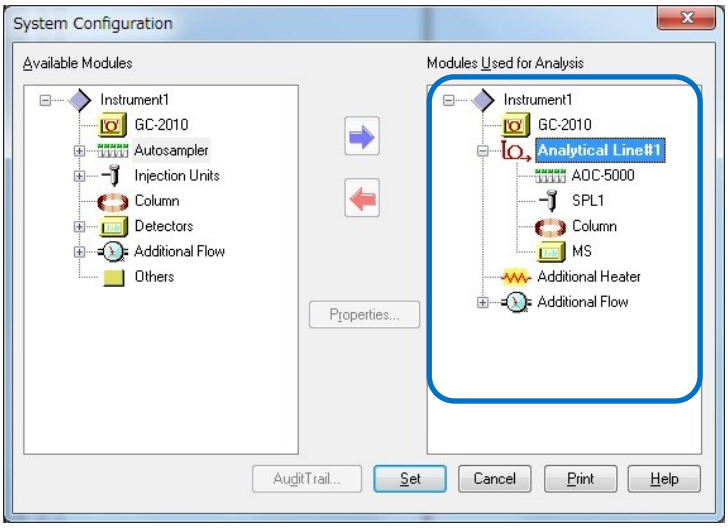


2.3.2 Using an AOC-5000 Plus/AOC-6000 Plus Autoinjector

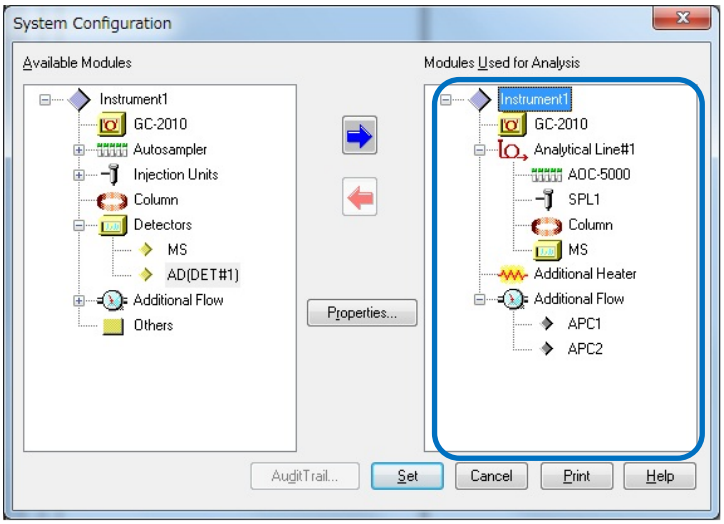
1) Install a liquid injection syringe adapter (10 μL capacity) on the AOC-5000 Plus or AOC-6000 series. For instructions on installing the adapter, refer to "3.*4.1 Changing a Syringe Adapter*" in the AOC-5000 Plus Control Software Instruction Manual or "*4. PAL Tool in the "AOC-6000 Operating Guide*."

2) Specify modules in the [Modules Used for Analysis] list as shown below.

If a Sniffer is Not Used



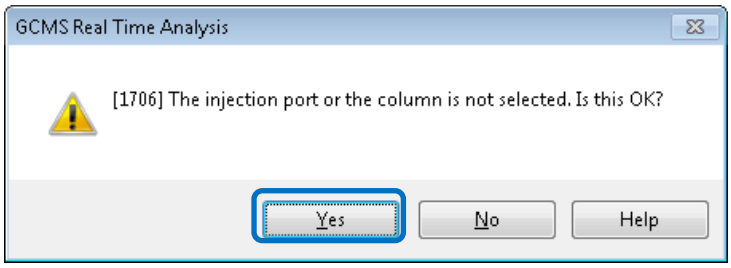
If a Sniffer is Used



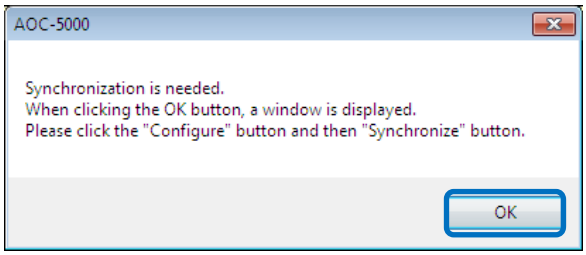
3) Click [Set].

NOTE

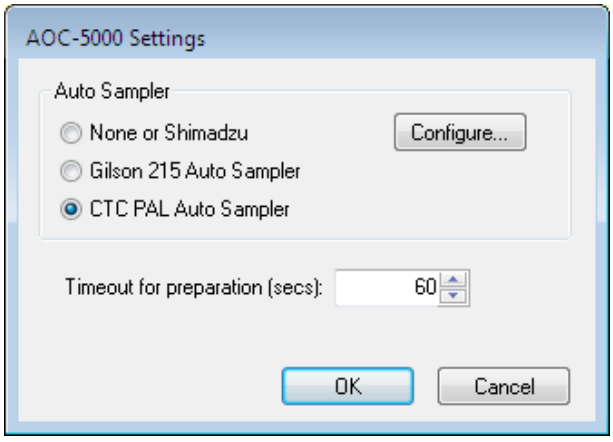
If a sniffer is used and the "[1706] The injection port or the column is not selected. Is this OK?" message is displayed, click [Yes].



4) If the following message appears, click [OK].



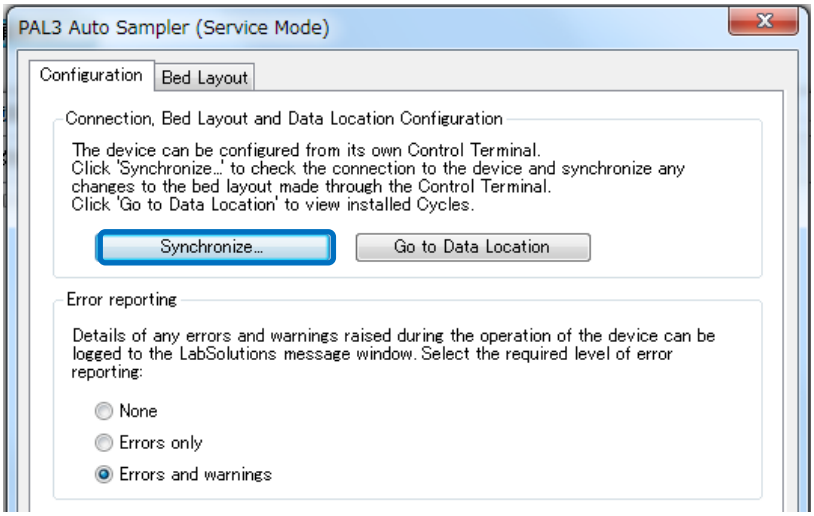
5) Click [Configure] in the [AOC-5000 Setting]/[AOC-6000 Setting] sub-window.［CTC PAL Auto Sampler］dialog is shown.



NOTE

Do not change the initial value of [Auto Sampler] and [Timeout for Preparation].Initial values are [CTC PAL Auto Sampler] and [60].

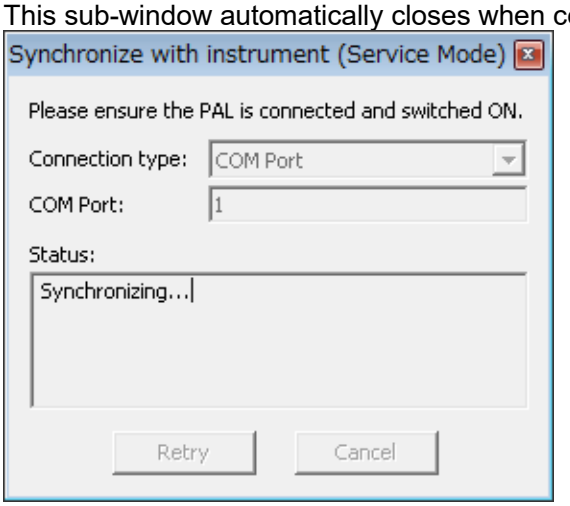
6) Click [Synchronize] on the [Configuration] tab page.
The [Synchronize with instrument] sub-window opens.



NOTE

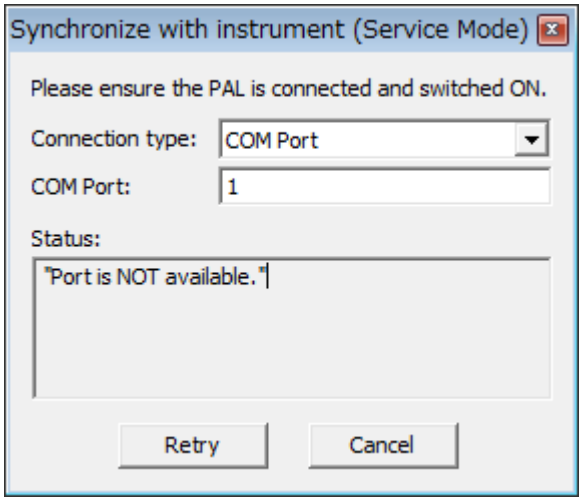
Select the [Errors and warnings] at the [Error reporting] in the [PAL3 Auto Sampler] sub-window. When not selected, it may cause that error and warning log is not saved correctly and data measurement doesn't stop.

7) This sub-window automatically closes when communication is successfully finished.



NOTE

If communication is not successful, the [COM Port] setting is enabled. If the communication cable (RS-232C) is connected to a COM port that is not COM1, enter the appropriate COM port number in the [COM Port] field and click [Retry]. Select a COM port number between 1 and 4. This software is only guaranteed to function properly using an RS-232C cable (via a COM port).



8) Click [OK] in the [PAL3 Auto Sampler] sub-window.

9) Click [OK] in the [Edit AOC-5000 Config] / [Edit AOC-6000 Config] sub-window.

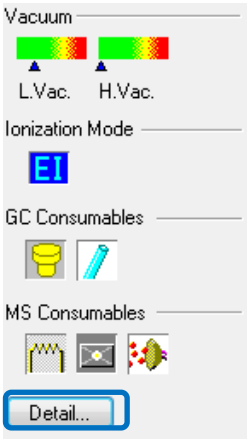
10) Confirm the AOC-5000 status in the instrument monitor.

NOTE

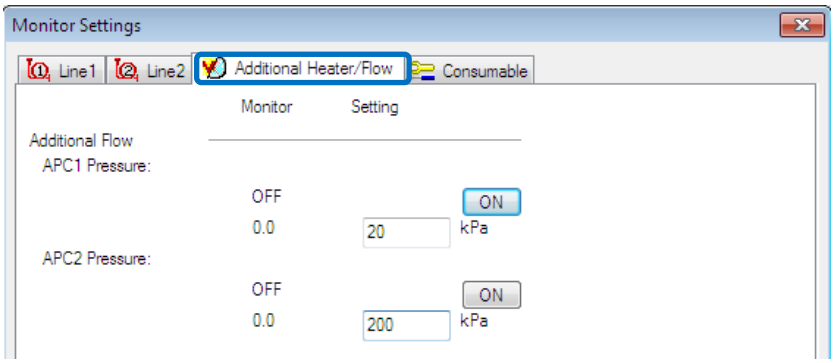
It takes about one minute until [AOC] changes to [Ready]. Once [AOC] changes to [Ready], go to the next step, even if [GC] and [MS] are [Not Ready].

11) If a sniffer is used, switch the APC pressure ON, as described in steps 12) to 14). If a sniffer is not used, skip to step 15).

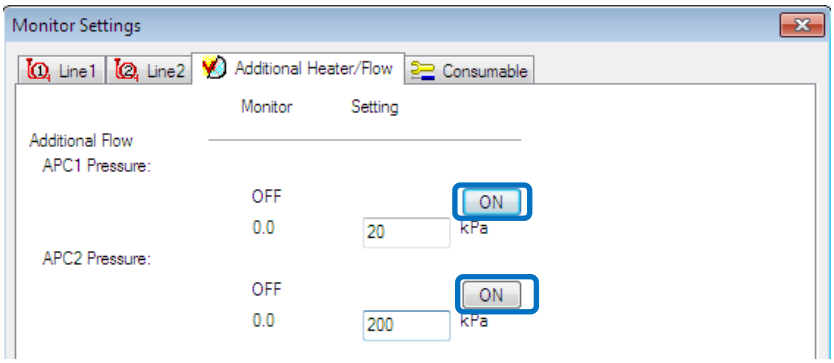
12) Click the [Detail].



13) Open the [Additional Heater/Flow] tab-page.



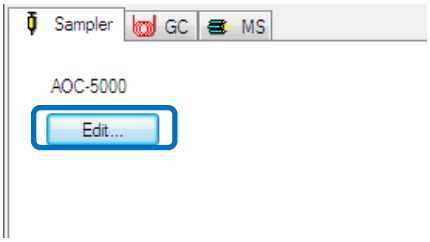
14) Click the [ON/OFF] switching button to change the status of [APC1] and [APC2] to [ON].



15) In the [Acquisition] window, open the method file located in the folder created in "*2.1 Preparation*".
Click the [Sampler] tab.

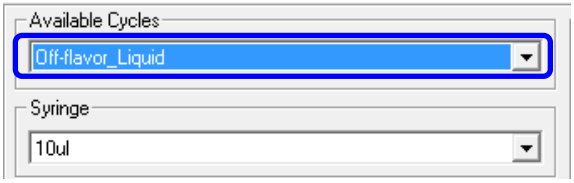
16) Click [Edit].

The [Instrument Method Editor] sub-window is displayed.



> **NOTE**
>
> If an AOC-6000 series is used for steps 17) to 21) below, refer to and follow the instructions in "*4. Method Settings*" of the AOC-6000 Control Software Instruction Manual. In addition, see [Liquid Injection] to specifying methods and "*Appendix 1. Analytical Conditions*" to specify parameters.

17) Select the [Off-flavor_Liquid] injection cycle at [Available Cycles].



18) Select [10μl] at [Syringe].

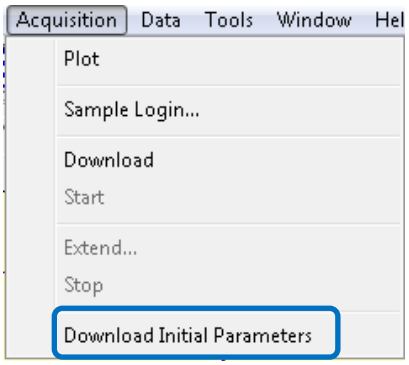
19) Click ✓ Apply to apply the parameter settings.

20) Click  to close the [Instrument Method Editor] sub-window.

21) Save the method file.

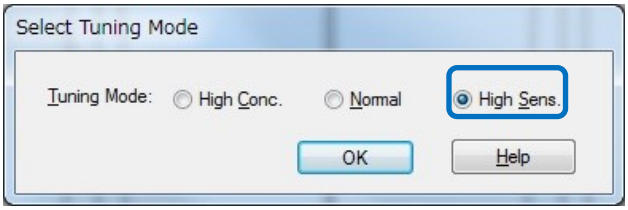
22) Repeat steps 15) to 21) for other method files in the folder created in "*2.1 Preparation*".

23) Click [Download Initial Parameters] on the [Acquisition] menu with the last method file loaded. Parameter settings are sent to the instrument



24) Wait until each parameter setting changes to the specified settings and the [GC] and [MS] settings change to [Ready].

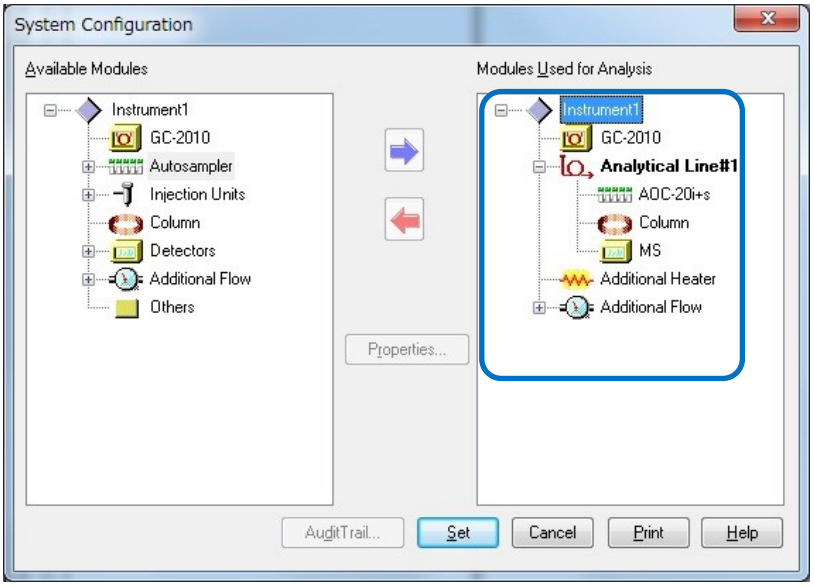
25) Perform auto-tuning, using the [High Sens.] tuning mode.



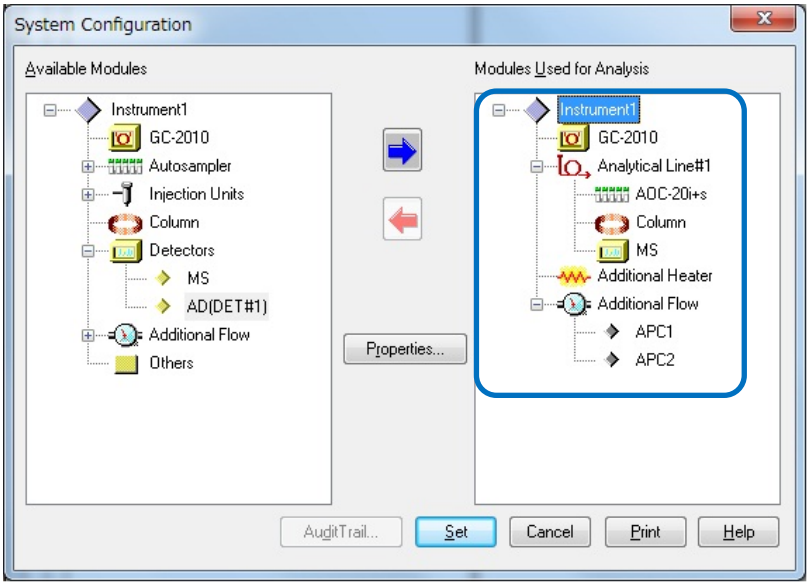
2.3.3 Using an OPTIC-4 Inlet

1) Specify modules in the [Modules Used for Analysis] list as shown below.

If a Sniffer Is Not Used.



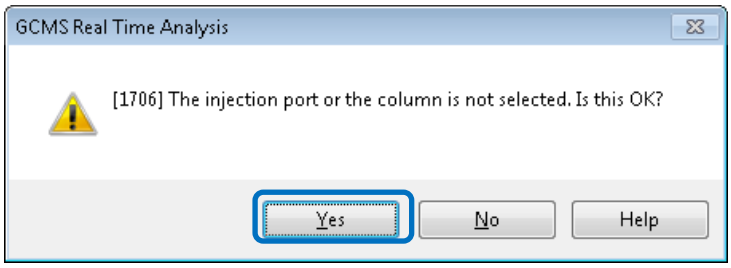
If a Sniffer Is Used.



2) Click [Set].

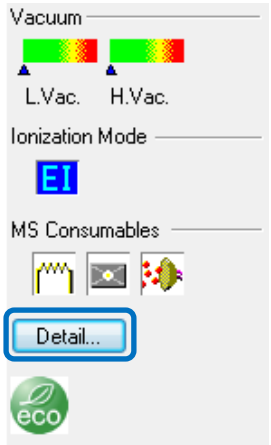
NOTE

If a sniffer is used and the "[1706] The injection port or the column is not selected. Is this OK?" message is displayed, click [Yes].

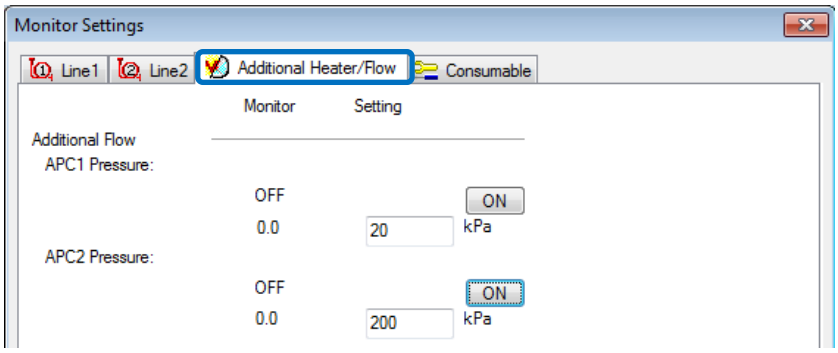


3) If a sniffer is used, switch the APC pressure ON, as described in steps 4) to 6). If a sniffer is not used, skip to step 7).

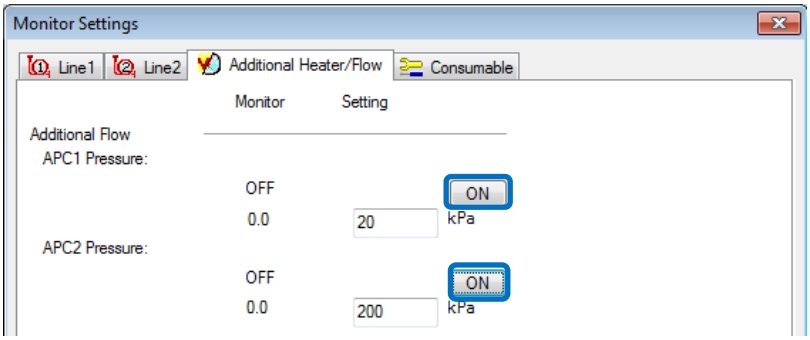
4) Click the [Detail].



5) Open the [Additional Heater/Flow] tab-page.



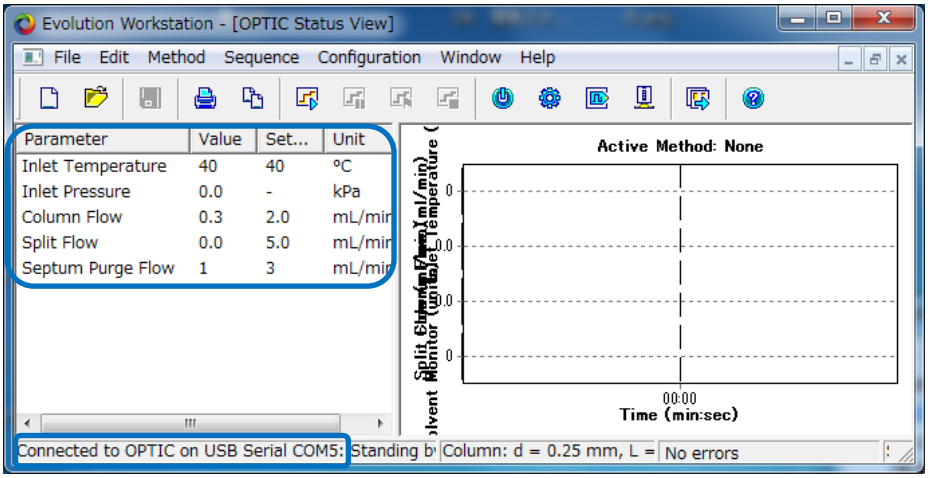
6) Click the [ON/OFF] switching button to change the status of [APC1] and [APC2] to [ON].



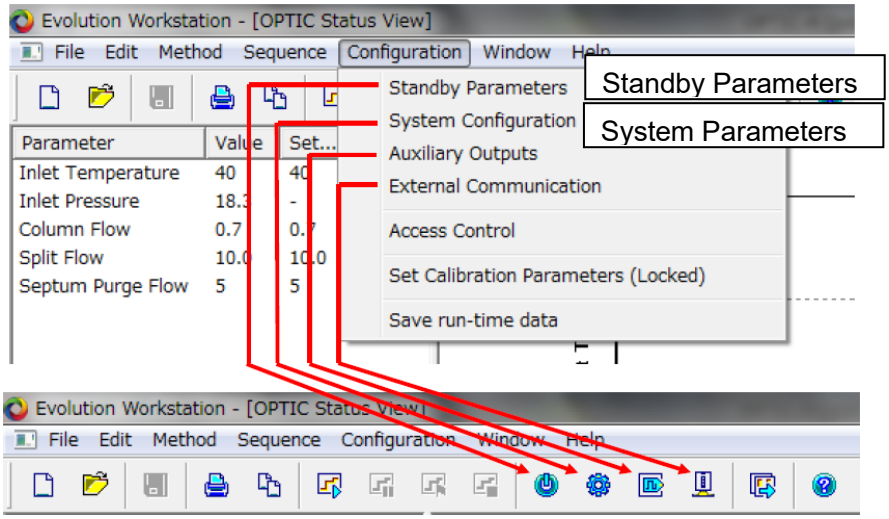
7) Check that the OPTIC-4 is turned ON.

8) Run the Evolution Workstation.

The [OPTIC Status View] sub-window is displayed. When communication is established between the computer and OPTIC-4 control unit, the standby status parameter setting values and current values are displayed in the [OPTIC Status View] sub-window. The system status display area indicates the connection method and port number used to connect to the computer.

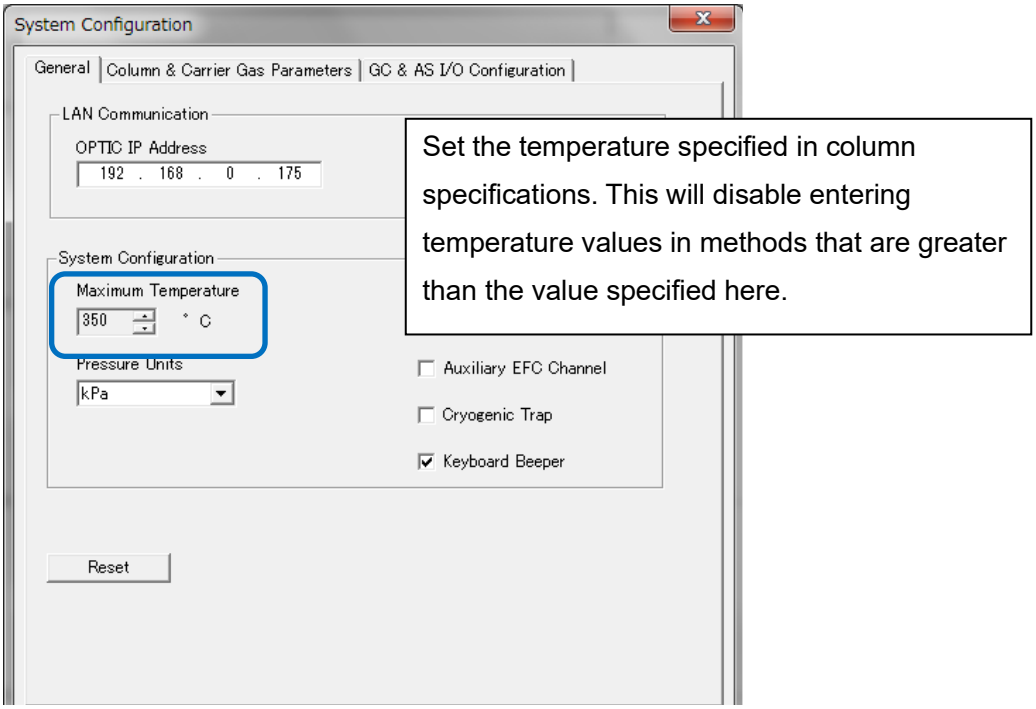


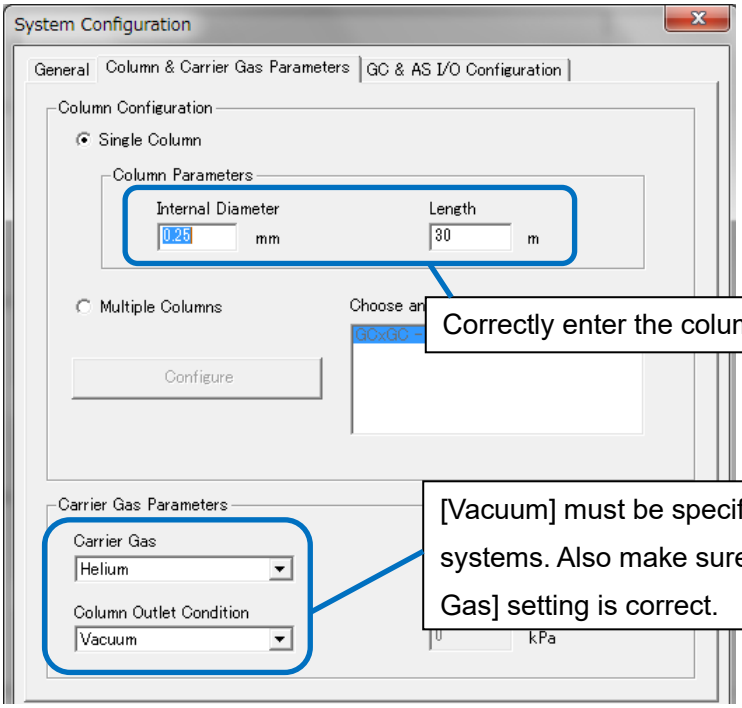
9) Configure the [Configuration]-[System Configuration] and [Standby Parameters] settings as shown below.



<u>System Configuration Settings</u>

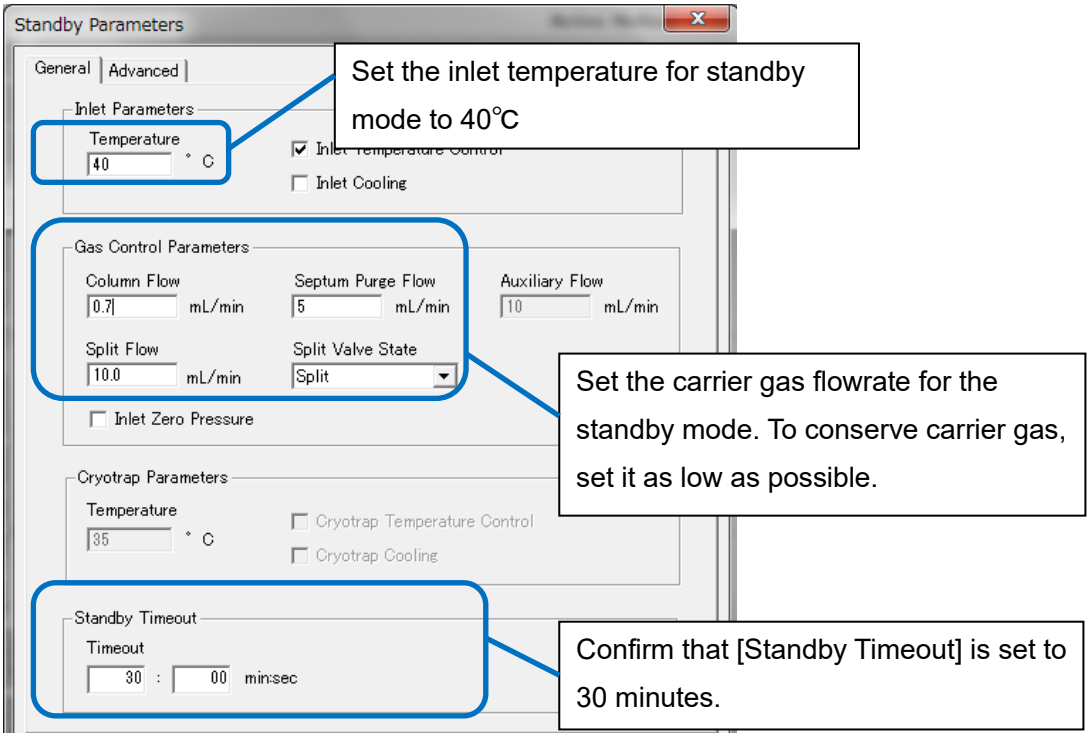
Set the parameter setting indicated with blue boxes as shown.





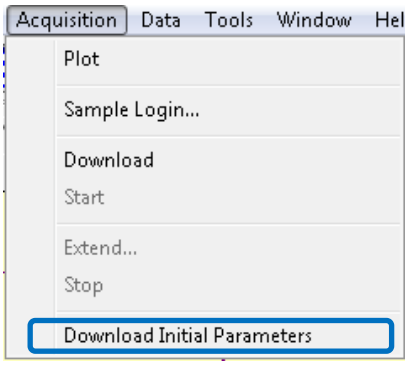
Standby Parameters Settings

Set the parameter setting indicated with blue boxes as shown.



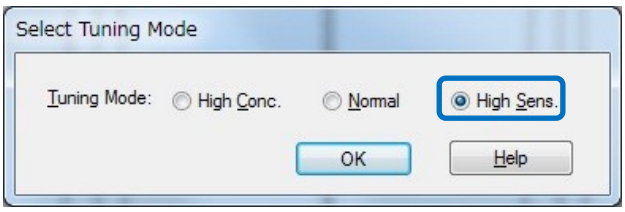
10) In the [Acquisition] window, open and overwrite the following method files, located in the folder created in "*2.1 Preparation*", to ensure configuration settings are consistent with the instruments that you use.

11) Click [Download Initial Parameters] on the [Acquisition] menu with the last method file loaded. Parameter settings are sent to the instrument.



12) Wait until each parameter setting changes to the specified settings and the [GC] and [MS] settings change to [Ready].

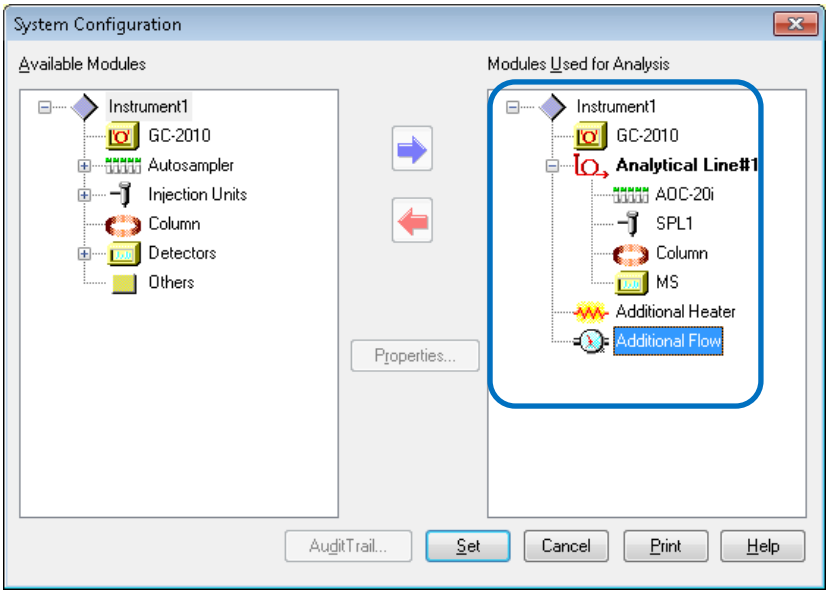
13) Perform auto-tuning, using the [High Sens.] tuning mode.



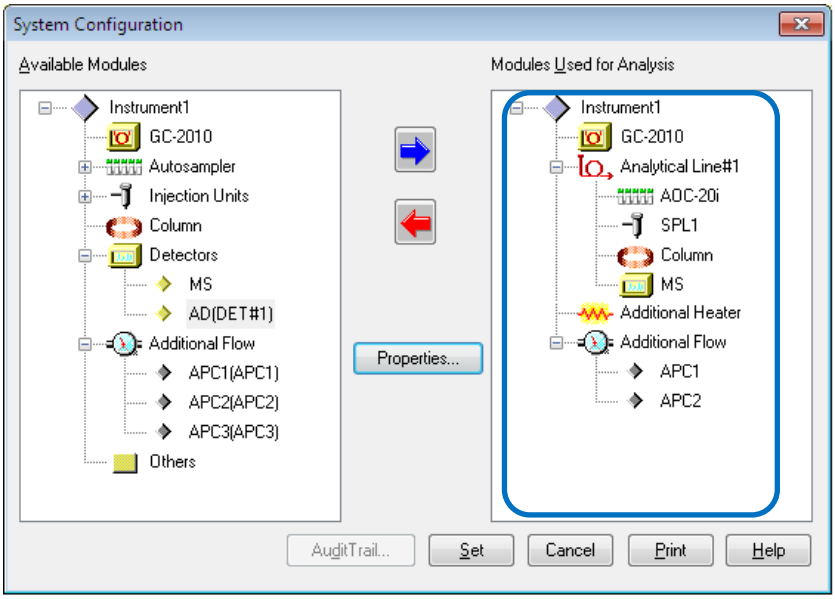
2.3.4 Using an TD-30 series

1) Specify modules in the [Modules Used for Analysis] list as shown below.

If a Sniffer is Not Used



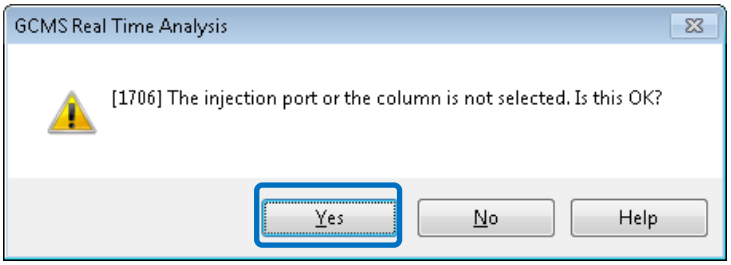
If a Sniffer is Used



2) Click the [Set] to complete the environment configuration

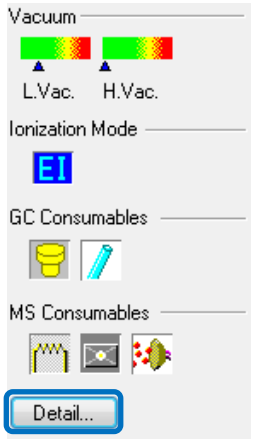
NOTE

If a sniffer is used and the "[1706] The injection port or the column is not selected. Is this OK?" message is displayed, click [Yes].

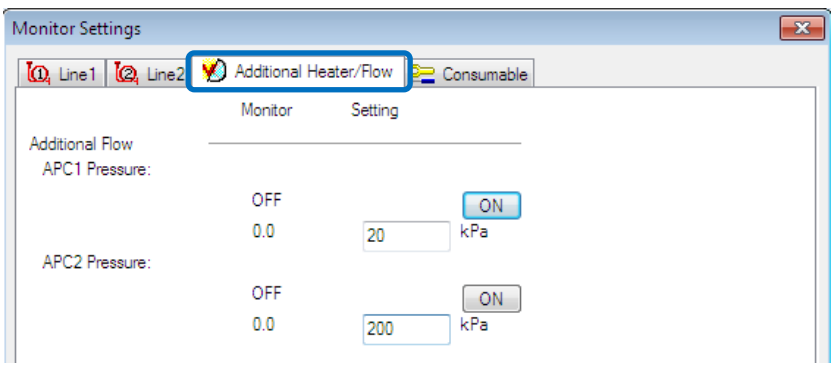


If a sniffer is used, switch the APC pressure ON, as described in steps 3) to 5). If a sniffer is not used, skip to step 6).

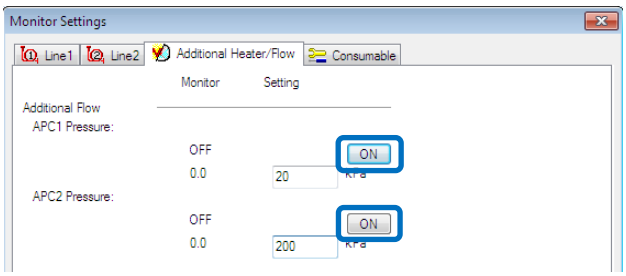
3) Click [Detail].



4) Click the [Additional Heater/Additional Flow] tab.



5) Click the ON/OFF button to switch [APC1] and [APC2] to [ON].



6) In the [Acquisition] window, open and overwrite the following method files, located in the folder created in "*2.1 Preparation*", to ensure configuration settings are consistent with the instruments that you use.

Method files for adjusting retention times

"SQ_MS_(column name)_AART.qgm" or "SQ_Sniff_(column name)_AART.qgm"

Method file for analyzing accuracy control

"SQ_MS_(column name)_QC.qgm" or "SQ_Sniff_(column name)_QC.qgm"

Method file for correcting calibration curve

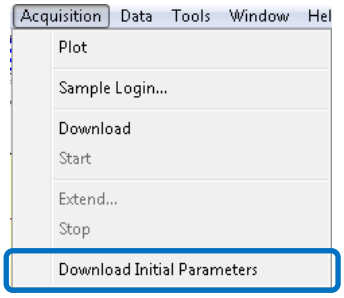
"SQ_MS_(column name)_Correct.qgm" or "SQ_Sniff_(column name)_Correct.qgm"

Method file for Template

"SQ_MS_(column name)_Template.qgm" or "SQ_Sniff_(column name)_Template.qgm"

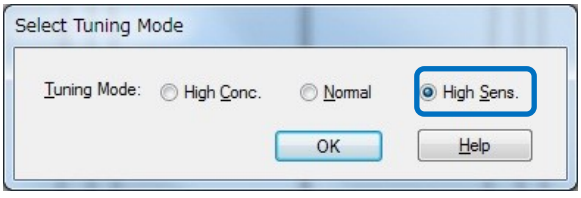
7) If using the GCMS-QP2020 NX or GCMS-TQ8040/8050 NX series, set the GC parameters listed in "Appendix 1 Analysis Conditions" in the four method files listed above.

8) Click [Download Initial Parameters] on the [Acquisition] menu with the last method file loaded. Parameter settings are sent to the instrument.



9) Wait until each parameter setting changes to the specified settings and the [GC] and [MS] settings change to [Ready].

10) Perform auto-tuning, using the [High Sens.] tuning mode.



2.4 Measuring Standard Samples

This section describes measuring the following three types of standard samples and performing various types of correction and instrument performance evaluation.

- For retention time adjustment: Standard n-alkane mixture sample
- For instrument performance evaluation: Standard mixture sample of four types of off-flavor components
- For calibration curve correction: Standard mixture sample of 4-bromofluorobenzene, 1,2-dichlorobenzene-d4, and acenaphthene-d10

Retention time adjustment : Measure the mixed n-alkane standard sample in order to correct the retention times registered in Smart Database. Correct the retention times for the target compounds based on the n-alkane identification results and retention indice of target compounds.(AART)

Instrument performance evaluation: To ensure high reliability for identification and quantitative analysis, it is important that the instrument is operating at a performance level above a certain level. Therefore, the operating status is verified by measuring a sample for evaluating instrument performance. For a description of the evaluation, see "*2.5.2 Evaluating Instrument Performance*". For instructions on preparing the standard mixture sample, see "*Appendix 3.2 Preparing Standard Samples for Evaluating Instrument Performance*".

Calibration curve correction: Calibration curve information calculated using the internal standard method is registered in the Smart Database. In general, calibration curve corrections must be calculated from a known quantity of an internal standard substance added to the sample, but adding an internal standard substance to the sample can affect sensory evaluations using the sniffer. Therefore, a function has been added to automatically correct calibration curves based on data from measuring calibration curve correction samples in advance. Samples for correcting calibration curves are prepared as a standard mixture sample of the above components (100 ng/mL concentration). For instructions on preparing the standard mixture sample, see "*Appendix 3.3 Preparing Standard Samples for Correcting Calibration Curves".*

Hint

To ensure high reliability for identification and quantitative analysis, it is recommended that a standard sample is measured about once every two weeks.

2.4.1 Measuring Standard Samples

> **Hint**
>
> For more information about preparing various standard samples, see Appendix 3 Standard Sample Preparation Method.

1) Place a standard n-alkane mixture sample (50 µg/mL) in position 1 of the autosampler.
2) Place a sample for instrument performance evaluation in position 2 of the autosampler.
3) Place a sample for calibration curve correction (100 ng/mL) in position 3 of the autosampler.
4) Open the batch file located in the folder created in 2.1.

	Vial#	Sample Name	Sample ID	Sample Type	Analysis Type	Method File
1	1	Alkane		0:Unknown	IT QT	SQ_MS_5MS_AART.qgm
2	2	QC_Sample		0:Unknown	IT QT	SQ_MS_5MS_QC.qgm
3	3	Correct_CAL_STD		0:Unknown	IT QT	SQ_MS_5MS_Correct.qgm

> **NOTE**
>
> If a TQ series system is used, the SIM and MRM analysis modes can be used for actual sample analysis. Correcting calibration curves requires acquiring calibration curve correction data that is consistent with the given analysis mode. Therefore, open the batch file for either the SIM or MRM analysis mode.
>
> ・ SIM mode analysis:
>
> TQ_(MS/Sniff)_(column name)_QC_SIM.qgb
>
> ・ MRM mode analysis:
>
> TQ_(MS/Sniff)_(column name)_QC_MRM.qgb

5) If an AOC-5000 Plus autoinjector is used, enter the tray name in the [Tray] column. If an AOC-6000 unit is used, enter the rack name in the [Tray] column. This setting is case specific. Make sure it is set correctly.

AOC-5000 Plus

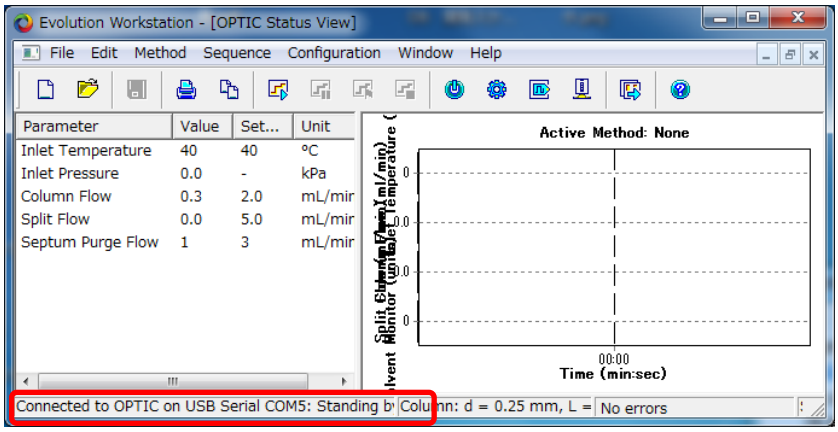
Tuning File	Data Descr	Tray
		Tray1

AOC-6000 Plus

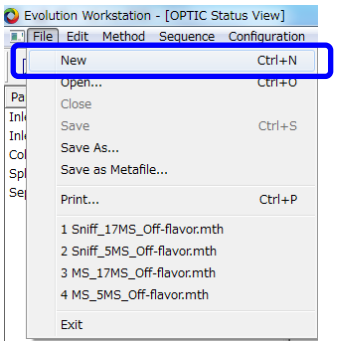
Tuning File	Data Descr	Tray
		Rack 1

6) Start the realtime batch analysis.
   If an OPTIC-4 inlet is used, create OPTIC-4 methods as described in steps 7) to 20). If an OPTIC-4 inlet is not used, skip to step 21).

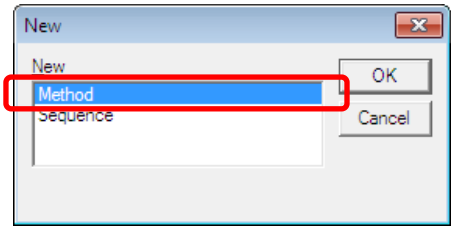
7) Confirm that the system status indicated in the [OPTIC Status View] sub-window of the Evolution Workstation is "Standing by."



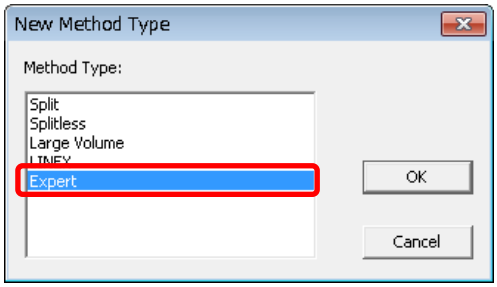
8) Click (New) on the toolbar.



9) Select [Method].

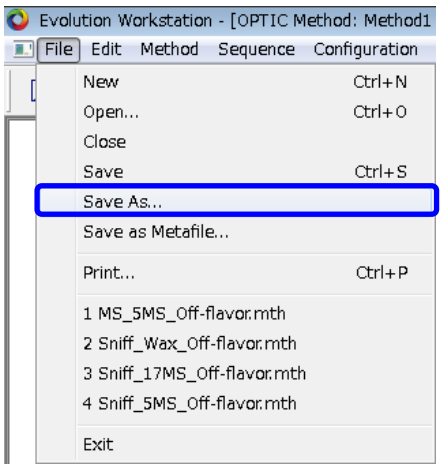


10) Select [Expert].



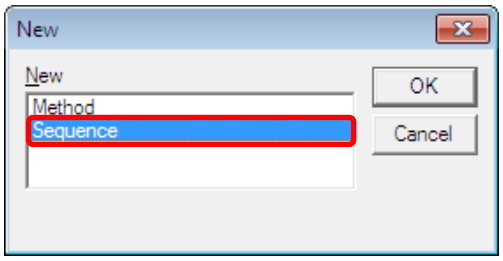
11) Specify parameter settings in the method file. For setting values, see "*6. OPTIC-4 Parameters in Appendix 1. Analytical Conditions*".

12) Click [Save as] on the [File] menu to save the method.

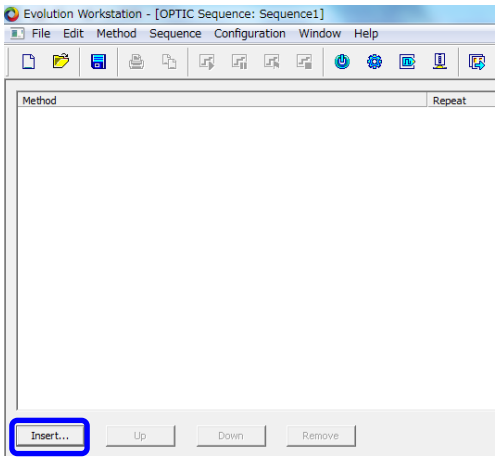


13) Select [New] on the [File] menu.

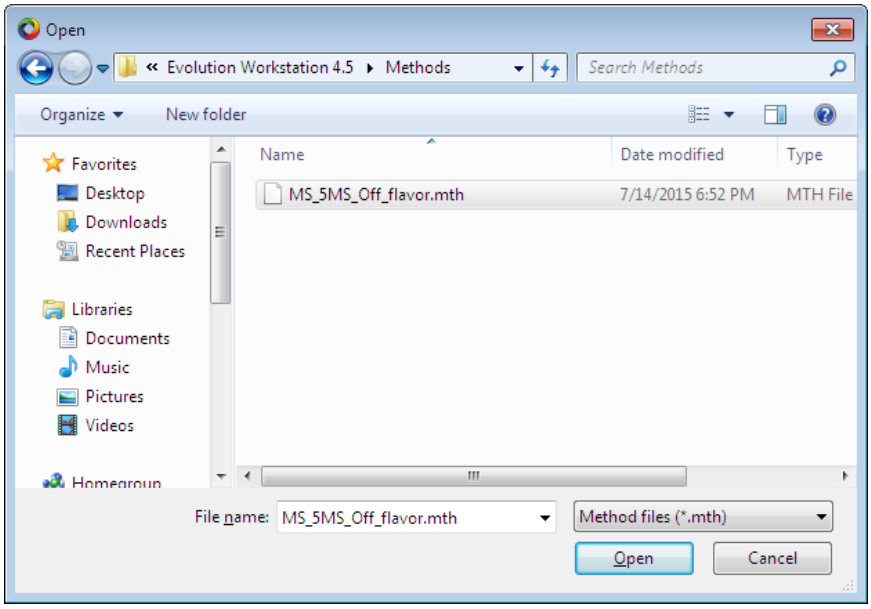
14) Select [Sequence].



15) Click [Insert].

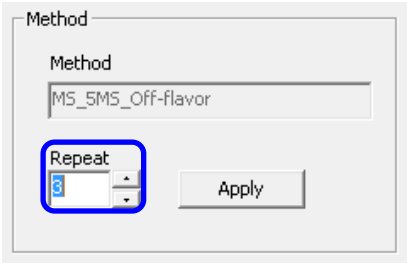


16) Select the OPTIC-4 method file saved in step 12).



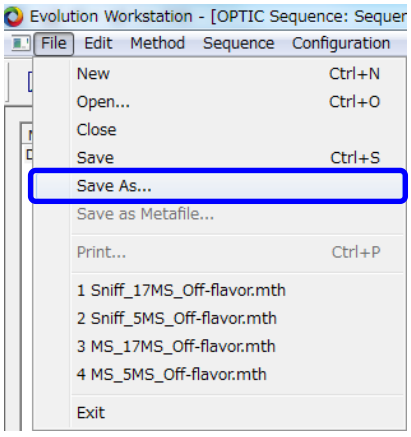
17) Specify how many times to repeat the analysis in [Repeat].

Specify the same repeat value as the repeat value executed for realtime batch analysis.



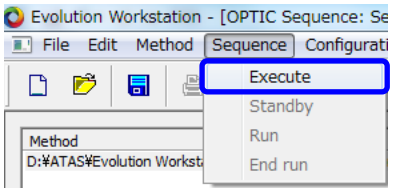
18) Click Apply.

19) Click [Save as] on the [File] menu to save the sequence.



20) Select [Execute] on the [Sequence] menu.

The system prepares for injecting samples and then automatically starts the analysis.

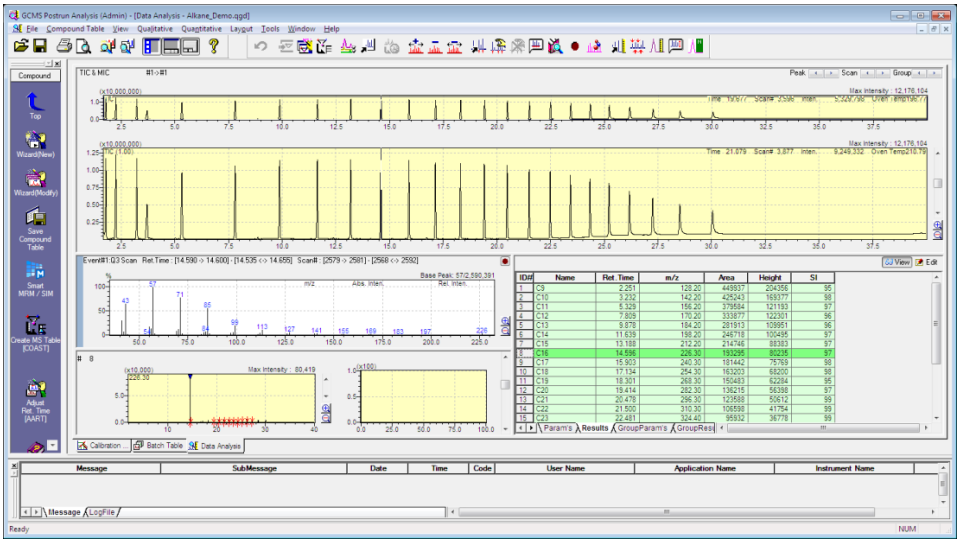


21) Starts the analysis automatically.

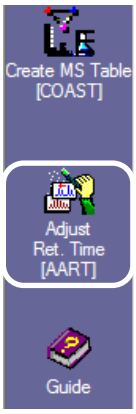
＜Confirming Off-flavor Using a Sniffer＞

Using a sniffer allows you to confirm the given off-flavor can actually be smelled. In that case, we recommend remeasuring the sample use for evaluating the instrument performance.

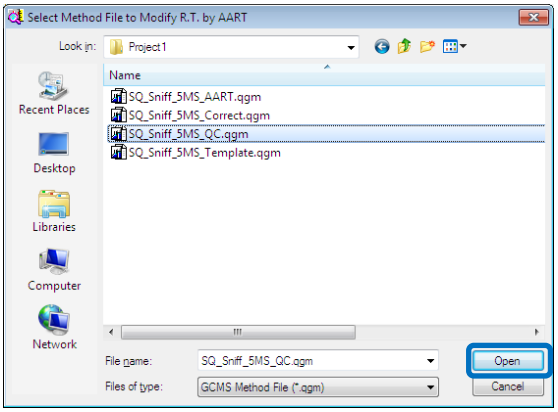
1) Load the data file for Retention time adjustment acquired in "*2.4 Measuring Standard Samples*".
2) Check and correct the identification results. If the sample has been misidentified, perform either manual identification or manual peak integration to identify it correctly.



3) Click the [ARRT] icon on the compound tab.

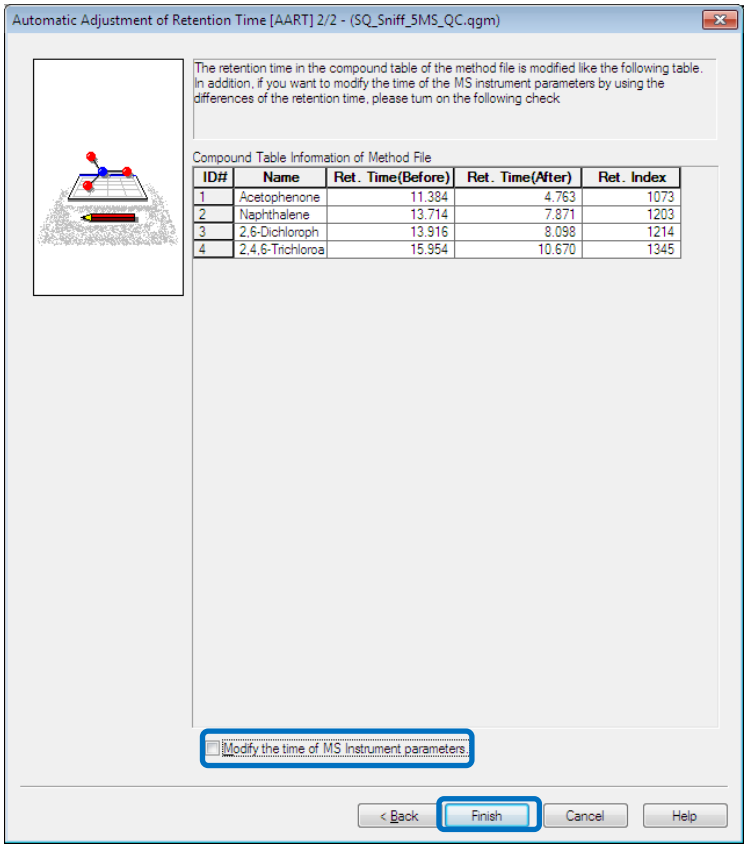


4) Select the method file for evaluating performance and then click [Open].



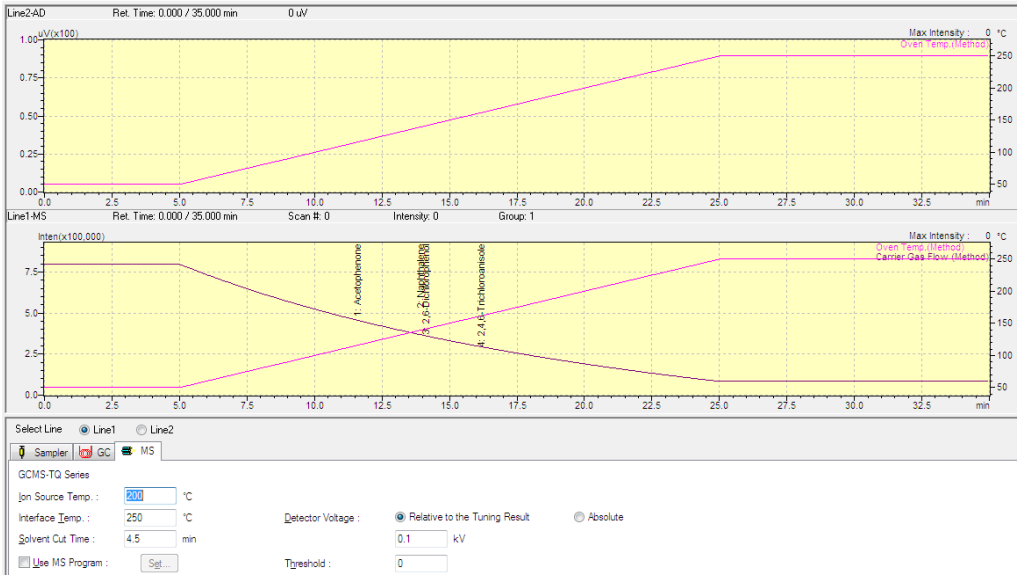
5) The [Automatic Adjustment of Retention Time [AART]1/2] window is displayed. Click [Next].

6) **Clear** the [Modify the time of MS Instrument parameters.] checkbox. Then click [Finish].



7) Save the method file.

8) Measure the sample for evaluating instrument performance. When the compound name is displayed in the analysis sub-window for the retention times of eluted components, smell the corresponding odor near that point.



9) Confirm that at least one of the four types of compounds can be smelled.
The smell of trichloroanisole is especially easy to confirm.

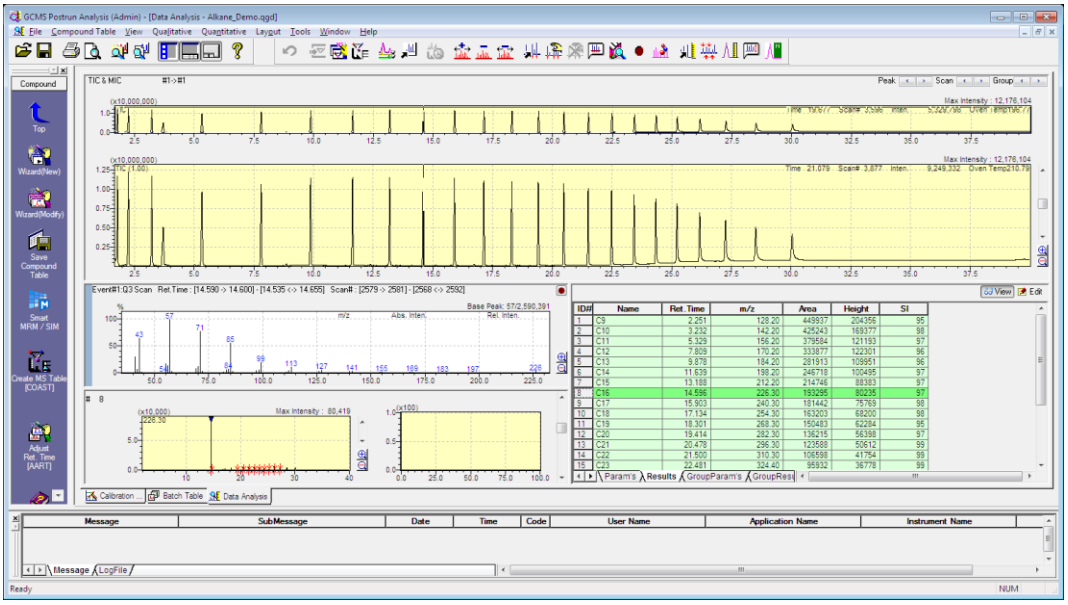
> **NOTE**
>
> Smelling the odor only provides a guideline. Due to individual differences in the threshold of when we can smell compounds, it may not be possible to smell the odor even when the system is functioning properly.

2.5 Analyzing Data from Standard Samples

2.5.1 Identifying n-Alkanes

1) Load the data file for Retention time adjustment acquired in "*2.4 Measuring Standard Samples*".

2) Check and correct the identification results. If the sample has been misidentified, perform either manual identification or manual peak integration to identify it correctly.



3) After the analysis, click the  (Save) icon on the toolbar. The identification results are saved to the data file.

2.5.2 Evaluating Instrument Performance

1) Check the following items in the results report output after the realtime batch analysis was executed in "*2.4 Measuring Standard Samples*".

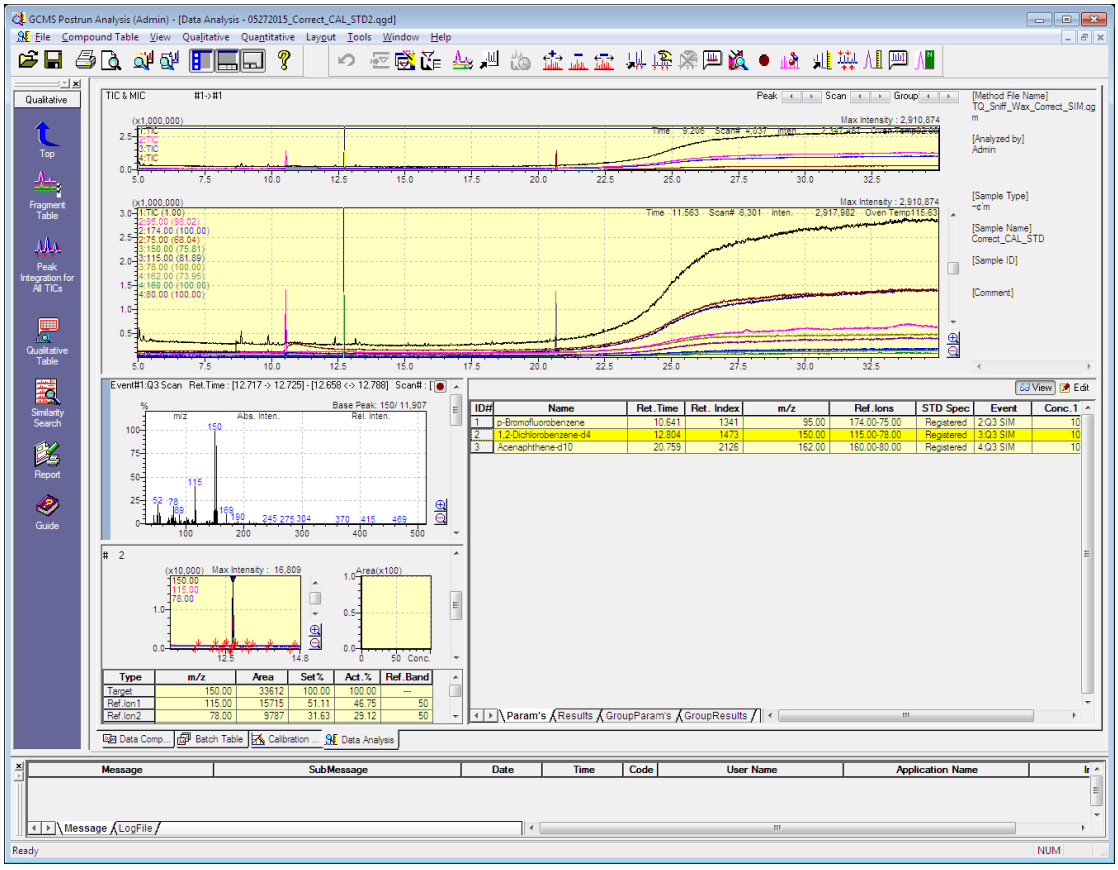
Content and Criteria for Evaluating Instrument Performance

Evaluation No.	Evaluation item	Criteria	Non-Passing Result
1	Instrument sensitivity	All compounds are identified. (Check that each peak top is marked with a circle.)	Clean the ion source and replace the insert.
2	Tailing factor	Tailing factor for 2.6-dichlorophenol is 2.5 or lower.	Cut 50cm from the injection unit end of the existing column or replace the column
3	Mass spectral pattern	Similarity for acetophenone is at least 60.	Perform tuning again and then remeasure.

2.5.3 Identifying Components in Samples for Calibration Curve Correction.

1) Load the data file for calibration curve correction samples acquired in "*2.4 Measuring Standard Samples*".

2) Check and correct the identification results for each component. If the sample has been misidentified, perform either manual identification or manual peak integration to identify it correctly.



3) After the analysis, click the (Save) icon on the toolbar. The identification results are saved to the data file.

Leave the data file open. It will be used again in "*2.6 Creating Method Files for Analyzing Samples*".

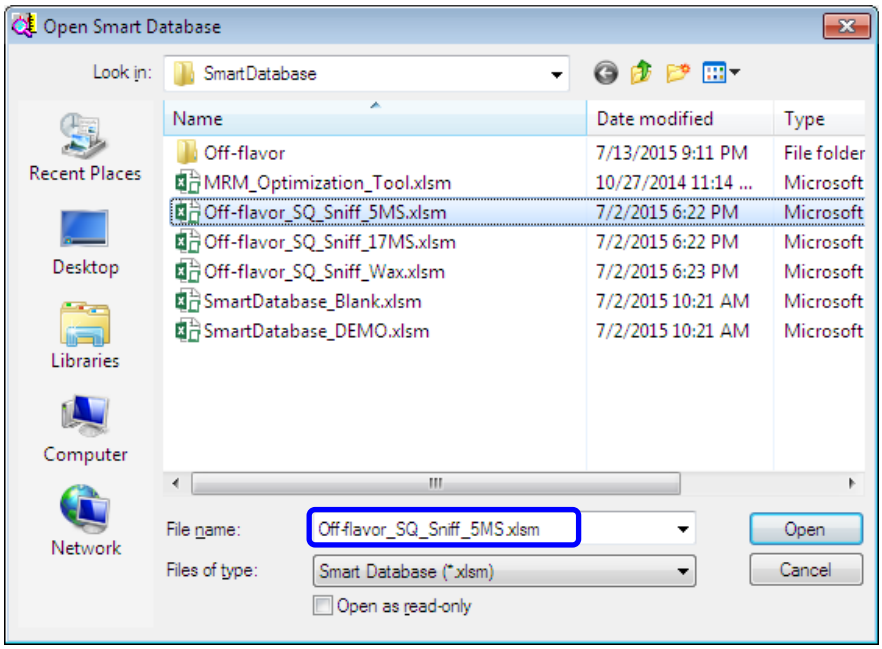
2.6 Creating Method Files for Analyzing Samples

1) Load the data file for Retention time adjustment acquired in "*2.4 Measuring Standard Samples*".

2) Click the 

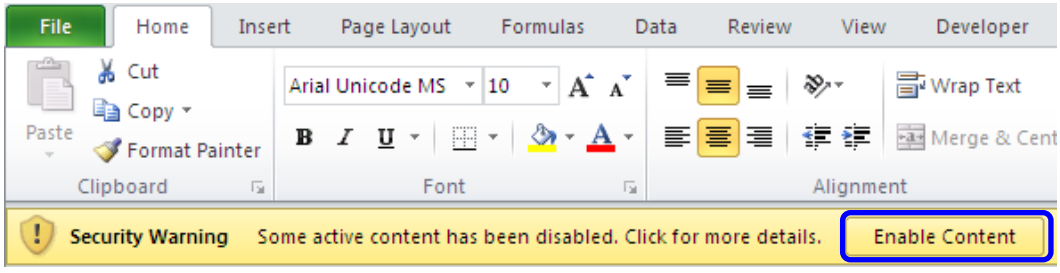
[Smart MRM / SIM] icon in the [Compound] assistant-bar.

3) Select the Smart Database file (Excel file) for the column used and then click [Open].
If using a sniffer, select the "Off-flavor_SQ_Sniff_(column name).xlsm" file.
If not using a sniffer, select the "Off-flavor_SQ_MS_(column name).xlsm" file.



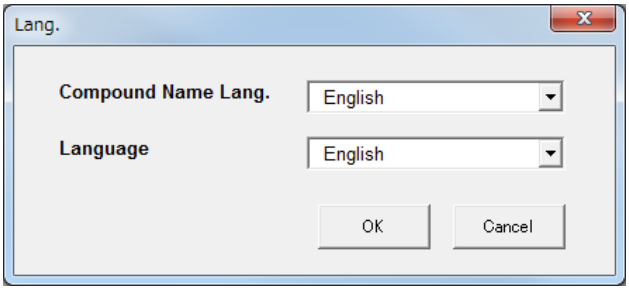
NOTE

If the following message is displayed on the message bar on Excel, Click [Enable Content].



Hint

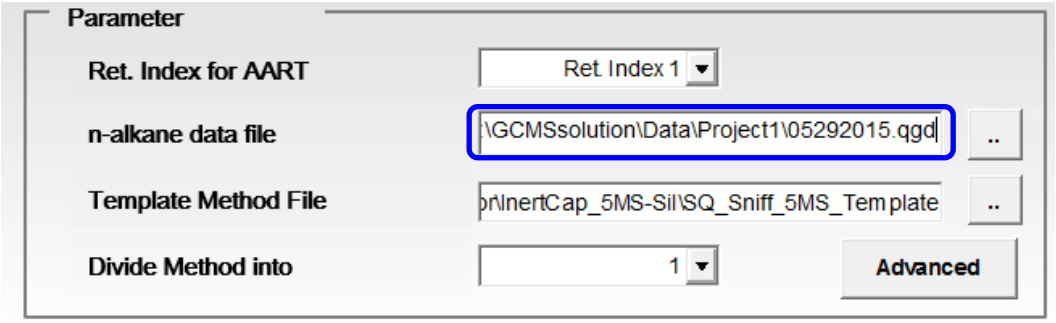
To change the display language, click [Lang.] and specify the language for displaying compound names and other text.



Compound Name Lang: Select the language for compound name notation. Select from Japanese and English.

Language: Select the display language for the Smart Database window.

4) Confirm that the alkane data file used in "*2.5.1 Identifying n-Alkanes"* is loaded.



NOTE

The optimal file for each database is set at [Template Method File]. Data acquisition and analysis conditions in the method file specified here are applied when a method is created.

5) If using a TQ series system, either the MRM or SIM measurement mode can be selected. For the SQ series, only SIM can be selected.

Serial#	Type	Acq. Mode	ISTD Group	Level1 Conc (IS)	Method No.	Compound Name (E)
1	Target	SIM			1	Propionic acid
2	Target	SIM			1	Pentanal
3	Target	SIM			1	Acetoin
4	Target	SIM			1	Methyl methacrylate
5	Target	SIM			1	Dimethyl disulfide
6	Target	SIM			1	Isobutyric acid
7	Target	SIM			1	sec-Butyl acetate
8	Target	SIM			1	1-Pentanol
9	Target	SIM			1	Toluene
10	Target	SIM			1	5-Hexene-2-one
11	Target	SIM			1	Butyric acid
12	Target	SIM			1	2-Hexanone

Hint

Under default conditions, three ions are selected. To change these, make your selections while checking the masses and ion ratios.

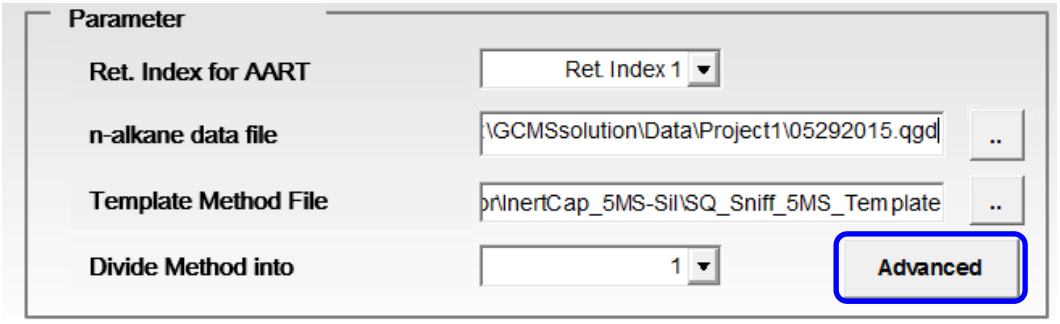
If you set the fields in the [Type] column to "T," it will be used as a target ion. If you set the fields in the column to "Ref.," it will be used as a reference ion. You can configure one target ion and up to five reference ions.

m/z for SIM or Scan

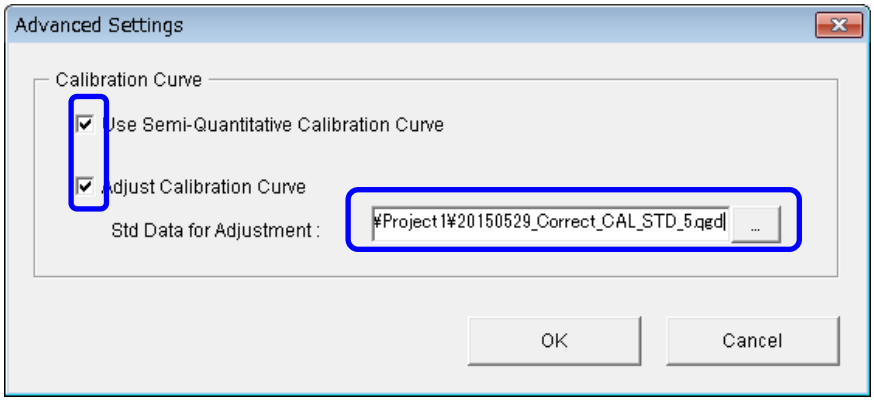
Ion1			Ion2			Ion3		
Type	m/z	Ratio	Type	m/z	Ratio	Type	m/z	Ratio
T	73.0	100.00	Ref.1	74.0	144.91	Ref.2	45.0	90.75
T	58.0	100.00	Ref.1	57.0			44.0	280.94
T	88.0	100.00	Ref.1	45.0			43.0	489.35
T	69.0	100.00	Ref.1	100.0			99.0	21.99
T	94.0	100.00	Ref.1	79.0			61.0	11.90
T	73.0	100.00	Ref.1	88.0			43.0	232.70
T	87.0	100.00	Ref.1	43.0			56.0	119.14
T	70.0	100.00	Ref.1	55.0	178.58	Ref.2	42.0	235.54
T	91.0	100.00	Ref.1	65.0	16.82	Ref.2	51.0	6.82
T	55.0	100.00	Ref.1	83.0	31.83	Ref.2	98.0	20.30
T	60.0	100.00	Ref.1	73.0	33.02	Ref.2	55.0	8.74

T
Ref.1
Ref.2
Ref.3
Ref.4
Ref.5

6) Click [Advanced].

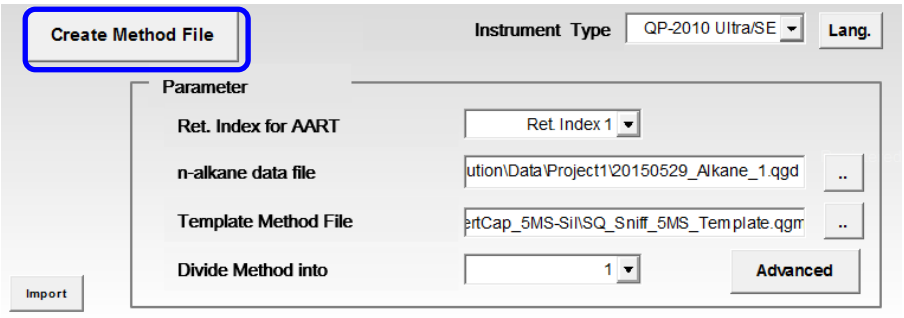


7) Select the [Use Semi-Quantitative Calibration Curve] checkbox.
Select the [Adjust Calibration Curve] checkbox and specify the data file that was already analyzed in "*2.5.3 Identifying Components in Samples for Calibration Curve Correction*".

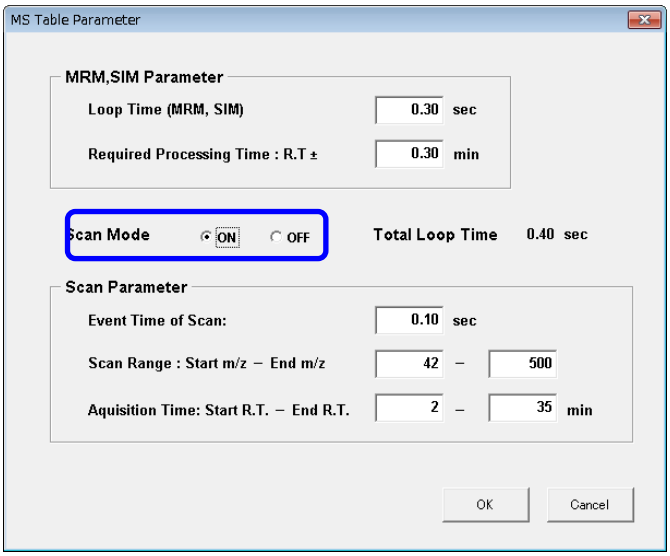


8) Click [OK] to close the sub-window.

9) Click [Create Method File]. The [MS Table Parameter] sub-window is displayed.



10) If the scan/MRM simultaneous analysis is used, select [ON] on the Scan Mode.



Hint

The MS table parameters are optimized, please change the parameters as needed.

- Loop Time (MRM and SIM)

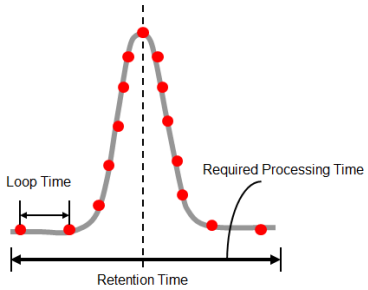
This is the sum total of the event times for all compounds configured to one group. If "0.3 sec" is entered, for example, data for each compound will be acquired every 0.3 seconds. To ensure good reproducibility, at least 10 data points must be acquired per peak. However, if the loop time is shortened, the time required to collect a single transition or ion will be shortened, reducing the sensitivity.

Hint

- Required Processing Time

This indicates the time range for measurements, centered on the retention time for each compound. For example, if "0.3 min" is entered, data will be acquired for each compound within a range of the retention time 0.3 min (i.e. for 0.6 minutes).

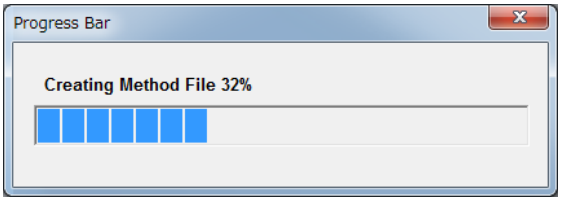
Measure a sample spiked with the standard sample using the method file created, and check for retention time offset. Pay attention to retention time offset due to impurities and to the peak widths (affected by tailing or other deformation), and reconfigure the required processing time accordingly. **Recommended values: 0.3 - 0.5 min**



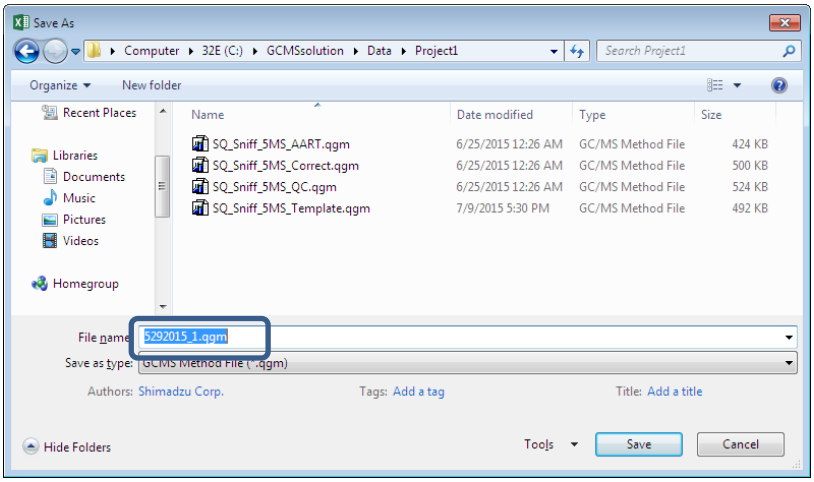
The required processing time specified here is applied to all compounds being measured. To specify required processing times individually for specific compounds, specify the values in cells AB to AC in the [Required Proc. Time for Each Comp.] column of the [Database] sheet. If the [Required Proc. Time for Each Comp.] column is blank, then the required processing time settings specified in the MS table sub-window are used.

Required Proc. Time for Each Comp. R.T. - X min	Required Proc. Time for Each Comp. R.T. + Y min
0.50	0.50

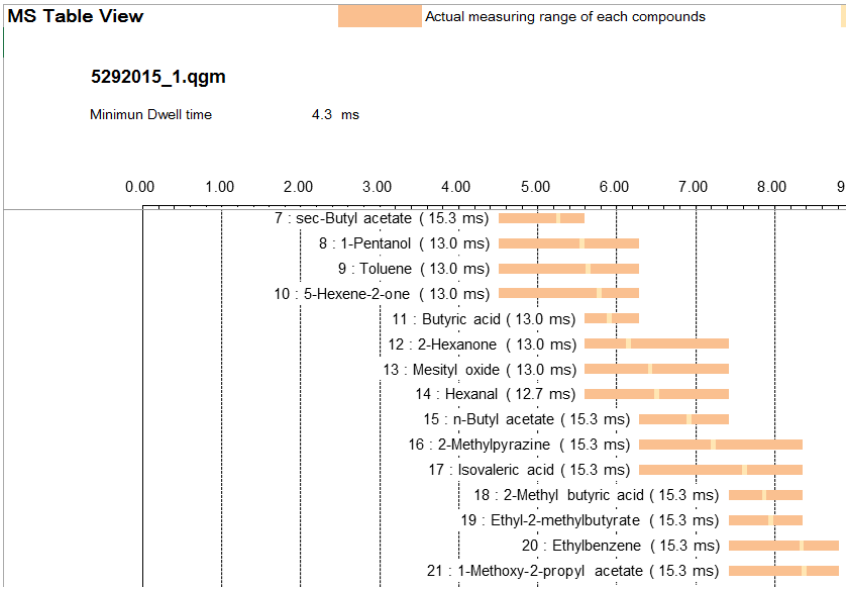
11) Clicking [OK] displays the [Progress Bar] sub-window and automatically creates method files.



12) Enter a file name, and then click [Save].

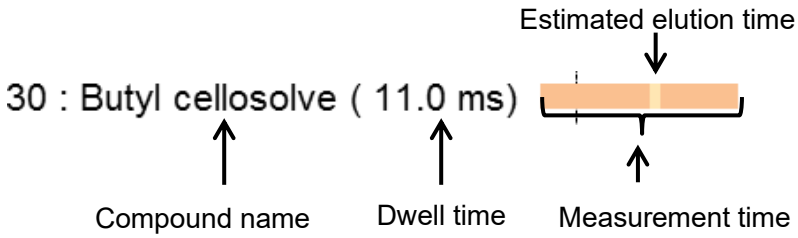


13) Check the time range for MRM measurements of target compounds.



Hint

You can use the [MSTableView] sheet to check the measurement time (hereinafter referred to as "dwell time") for each transition (ion) for each target compound.

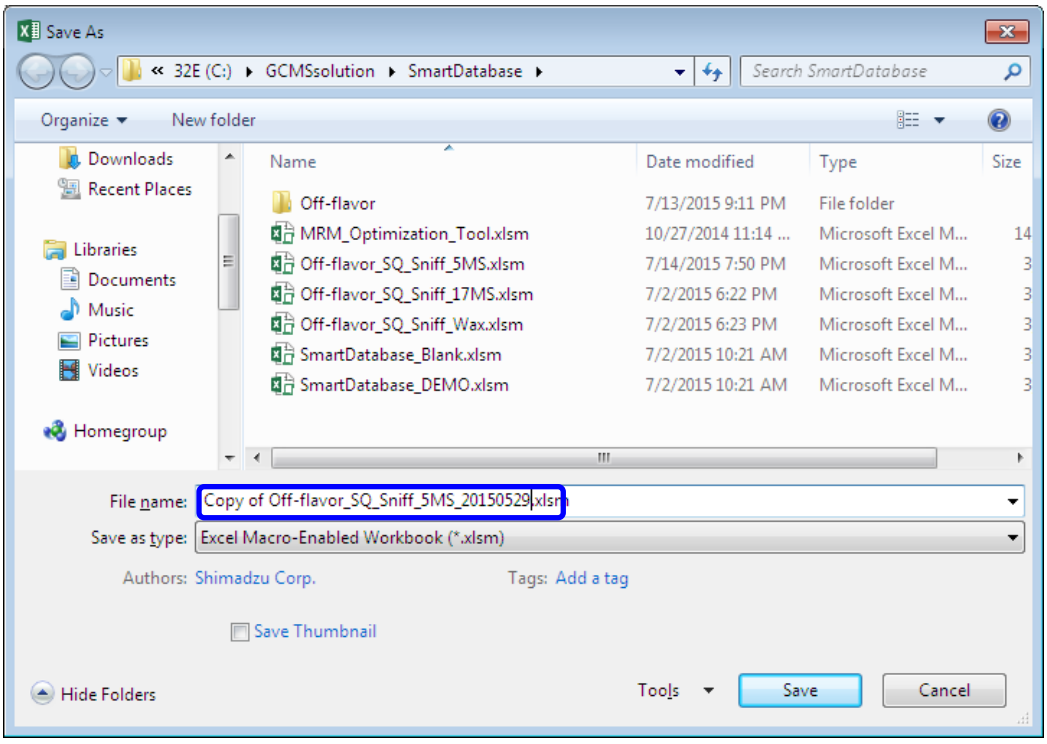


In addition, you can check the minimum value for the dwell time for all target compounds.

5292015_1.qgm

Minimun Dwell time 4.3 ms

14) Name the Smart Database file and save it.



Using AOC-5000 Plus or AOC-6000 series

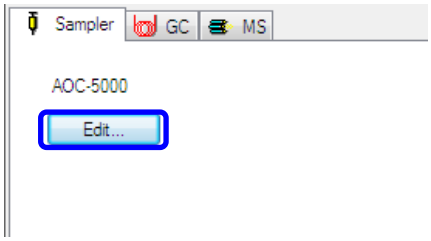
If an AOC-5000 Plus or AOC-6000 series is used, the injection cycle must be selected for the sample injection method used. After creating a method file from the database, perform the operations in the following procedure.

1) Install a syringe adapter, corresponding to the sample injection method to be used, on the AOC-5000 Plus or AOC-6000 series.

2) Open the method file saved in step 12) of "*2.6 Creating Method Files for Analyzing Samples*" in the [Acquisition] sub-window.

3) Click the [Sampler] tab.

4) Click [Edit].
The [Instrument Method Editor] sub-window is displayed.

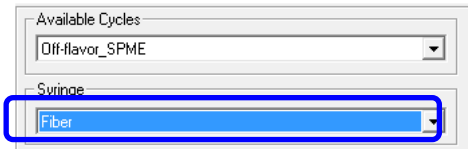


NOTE

If an AOC-6000 series is used for steps 5) to 11) below, refer to and follow the instructions in "*4. Method Settings*" of the AOC-6000 Control Software Instruction Manual. To specify parameter settings, see "*Appendix 1. Analytical Conditions.*"

5) Select the injection cycle at [Available Cycles].
Select the one of the following injection cycles based on the sample injection method used.
Off-flavor_Liquid (For liquid injection method)
Off-flavor_HS (For headspace method)
Off-flavor_SPME (For solid phase micro extraction method)

6) Select the syringe that is installed in the instrument at [Syringe].



7) Select the sample injection unit at [Cycle Arguments]-[Inject to].

Cycle Arguments

Parameter	Value
Pre Incubation Time (s)	300
Incubation Temp. (° C)	80
Pre Inc Agitator Speed (rpm)	250
Agitator On Time (s)	5
Agitator Off Time (s)	2
Needle Penetration (mm)	8
Fiber Penetration in mm	14
Extraction Time (s)	1800
Desorb to	GC Inj1
Injection Penetration (mm)	40
Desorption Time (s)	120
Post FibCondTime (s)	300
GC Runtime (s)	2700

8) Check the other parameter values for [Cycle Arguments] settings.
If the parameter settings are changed, make sure the setting is within the range appropriate for the given instrument. For more information about the parameter setting ranges, see Appendix—Method Parameters in the AOC-5000 Plus Control Software Instruction Manual.

9) Click ✓ Apply to apply the parameter settings.

10) Click  to close the [Instrument Method Editor] sub-window.

11) Save the method file.

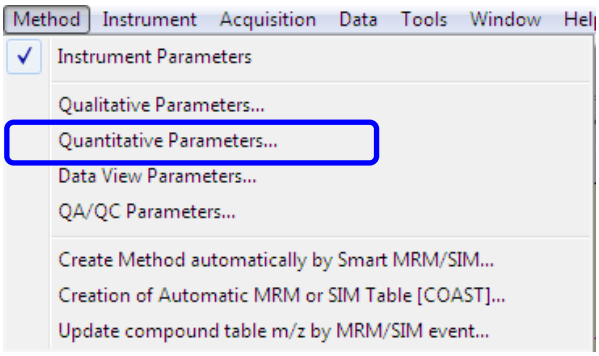
2.7 Analyzing Samples

Samples are analyzed using the method files created in "*2.6 Creating Method Files for Analyzing Samples*".

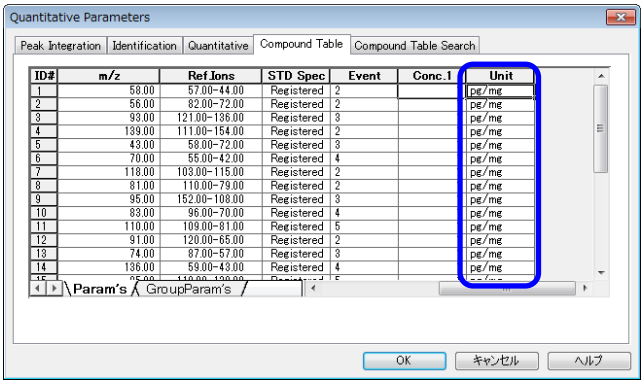
Hint

Semi-quantitative values calculated in analytical results are indicated as absolute values (pg) entered in the GC-MS(/MS) system. If the sample quantity and dilution factor are entered for realtime batch analysis, the software automatically calculates the concentrations in the sample. In that case, change the concentration units specified in the method file.

1) In the [GCMS Real Time Analysis] program, load the method files created in "*2.6 Creating Method Files for Analyzing Samples*".
2) Click [Quantitative Parameters] on the [Method] menu.



3) Click the [Compound Table] tab. In the [Unit] column, enter concentration units appropriate for the weight of sample acquired.
   Input example:
   Solid sample: pg/mg (sample acquisition quantity)
   Liquid sample: pg/μL (sample acquisition quantity)

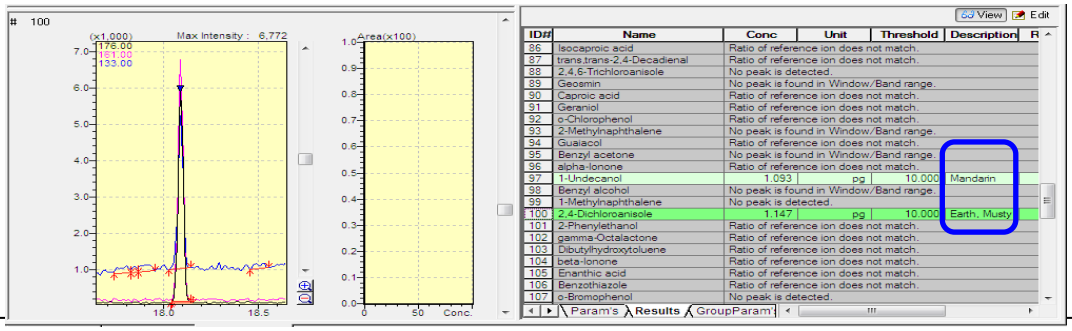


4) Click [OK] to save the method file.

Hint

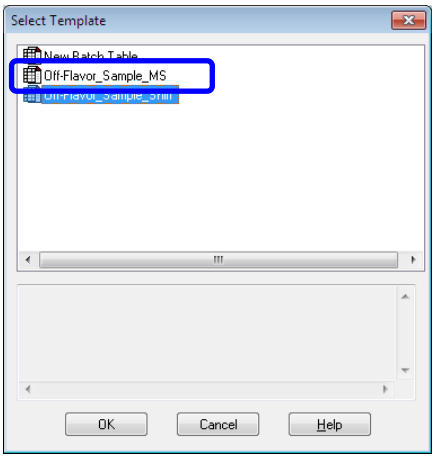
It is recommended that the smell of the actual abnormal odor sample be confirmed before analyzing samples.
As shown below, analytical results indicate sensory information for detected components. Confirming the smell of odors before analysis allows you to compare sensory information to those results.

Example: Analysis of sample with moldy odor
Smelling the odor sample before analysis allows you to confirm that it smells moldy. After analysis, the list of candidate odor-causing components can be refined by confirming the sensory information for detected components and extracting the components with a moldy odor.



2.7.1 Executing Realtime Batch Analysis

1) Place the measurement sample in the auto sampler.
2) Open the [batch table] window.
3) Click  (New) on the toolbar.
4) In the [File New] sub-window, select “Off-Flavor_Sample_MS”.



5) Specify [Vial#], [Sample Name], and [Sample ID] values. Also specify the method files created in "*2.6 Creating Method Files for Analyzing Samples*". To include automatically calculated component concentration values in quantitative results, specify [Sample Amt.] and [Dil. Factor] values

Vial#	Sample Name	Sample ID	Sample Am	Dil. Fact	Sample Type	Analysis Type	Method File
1	Sample_A	0001	2	100	0:Unknown	IT QT	5292015_1.qgm

Hint

Example of [Sample Amt.] and [Dil. Factor] Settings
The final quantitation value is calculated by dividing the concentration value determined from the calibration curve by the [Sample Amt.] value specified here. The final quantitation value is calculated by multiplying the concentration value determined from the calibration curve by the [Dil. Factor] value specified here.

[Sample Amt.]
Enter the weight of the sample acquired in terms of the concentration unit setting in the compound table.
Example: For a sample weighing 2.5 mg, enter "2.5."

[Dil. Factor]
This setting is mainly entered for solvent extracted samples injected as a liquid. Enter values as a ratio of final extract solution divided by sample injection volume.
Example: Assume a 2.5 mg solvent extracted sample is used to prepare 1000 μL of a final extract solution, from which 1 μL is injected into the GC/MS system.
Set [Sample Amt.] to "2.5" (mg) and [Dil. Factor] to "1000" (1000 μL/1 μL).

6) If an AOC-5000 Plus autoinjector is used, enter the tray name in the [Tray] column. If an AOC-6000 series is used, enter the rack name in the [Tray] column. This setting is case specific. Make sure it is set correctly.

AOC-5000 Plus

Report File	Tuning File	Data Descr	Tray
or\Off-flavor_Report.qgr			Tray1

AOC-6000 series

Report File	Tuning File	Data Descr	Tray
or\Off-flavor_Report.qgr			Rack 1

7) Delete any rows unnecessary for measurements by highlighting and right-clicking on the rows and clicking [Delete Row] on the right-click menu.
8) Name and save the batch file.
9) Execute the realtime batch analysis.

> **NOTE**
>
> If an OPTIC-4 unit is used, the OPTIC-4 method file must be executed when the realtime batch analysis is executed. Follow the instructions in steps 7) to 20) in "*2.4.1 Measuring Standard Samples.*"

2.8 Analyzing Measurement Data

2.8.1 Identigying Detected Components

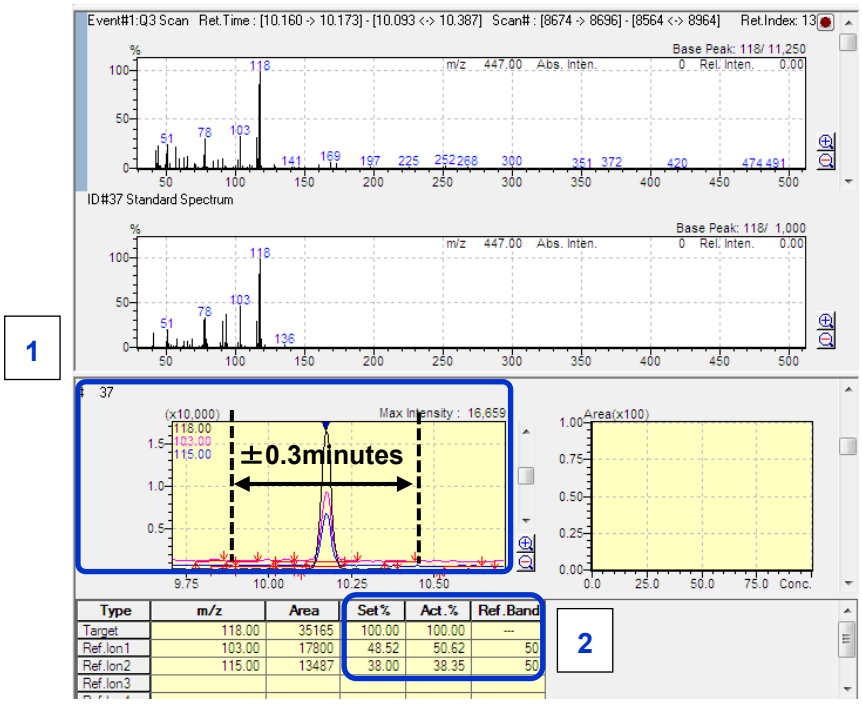
1) Run the [GCMS Postrun Analysis] program.

2) Open the measured data file.

3) Peak integration and peak identification processes are performed automatically based on the following criteria for compounds listed in the compound table. Check the identification results for each compound. If any are misidentified, identify them correctly either by manual identification or by manual peak integration.

Software Automatic Identification Criteria

1. Peak integration and peak identification in mass chromatograms.
   (Allowable range of registered retention times:±0.3 minutes)
2. Relative ion ratio for reference ions.
   (Allowable relative ion ratio range for registerd reference ions:±50%)

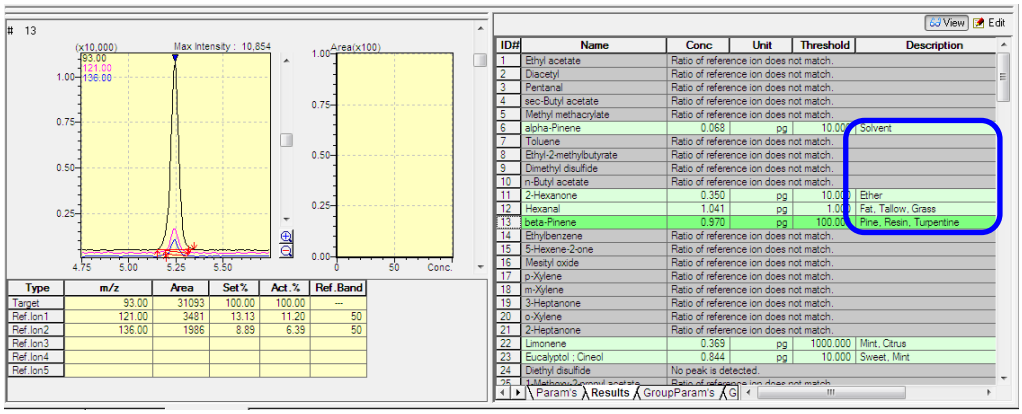


NOTE

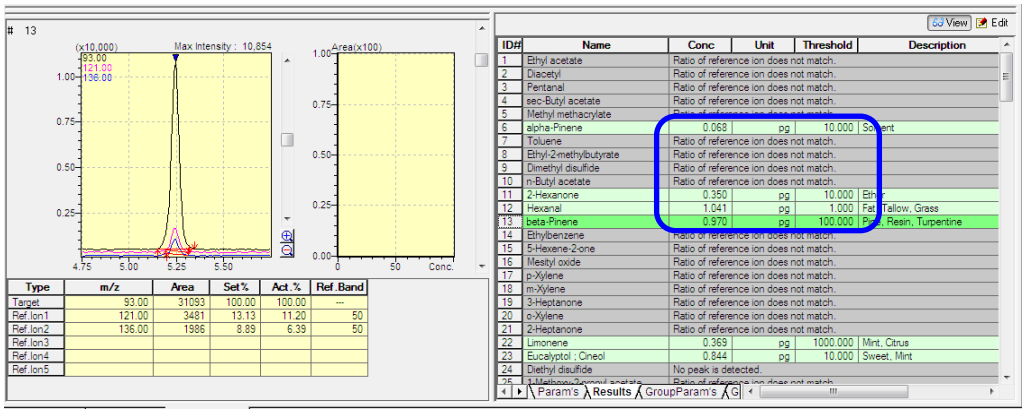
During automatic peak detection performed by GCMSsolution, peaks may be incorrectly identified or not identified under the following conditions. Check measurement results using similarity search on libraries or by checking with standard spectra and reference ion ratios registered in the method file.

- The concentration of the target component is extremely low.
- Ion of the target component overlaps with the sample-derived matrix.
- Instrument conditions are poor due to a contaminated glass liner, analytical column, or ion source.

4) If the off-flavor smell was confirmed before analyzing the sample, confirm the sensory information for the identified compounds. Sensory information is indicated in the [Description] column. The list of candidate odor-causing components can be refined by comparing the odor confirmation results to components with the same sensory information.



5) The concentration of identified compounds can be compared to corresponding odor threshold values to determine whether the identified components are present in concentration higher than the threshold value.



NOTE

- The concentrations calculated by this product are only approximate. It provides lower accuracy than conventional quantitative methods that involve preparing calibration curves. Furthermore, due to the differences in the instrument condition and extraction efficiency achieved by the sample pretreatment method used, the calculated quantitation values can differ significantly from the true values.
- The odor threshold values registered in the database were calculated by adding a standard sample to water. Therefore, depending on the sample, they can differ from precisely determined odor threshold values and only serve as reference information.

2.8.2 Comparing Multiple Sets of Data

Multiple sets of data can be compared. For example, this functionality is especially useful for comparing abnormal odor samples to normal samples. An overall comparison of chromatograms can be performed in the [Data Comparison] window. A comparison of specific components can be performed in the [Data Analysis] window.

Overall Comparison of Chromatograms

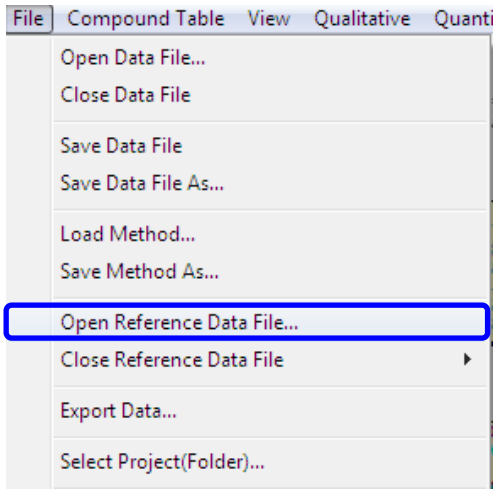
1) Click the [Data Comparison] icon on the [Postrun] assistant bar in the [GCMS Postrun Analysis] program.
2) Click  (Open) icon on the toolbar and select the data to compare.
3) This displays an overlay of the TIC chromatograms for the selected data, which allows you to compare differences in corresponding peaks in respective data.

Comparison of Specific Components

1) Open the [Data Analysis] window in the [GCMS Postrun Analysis] program

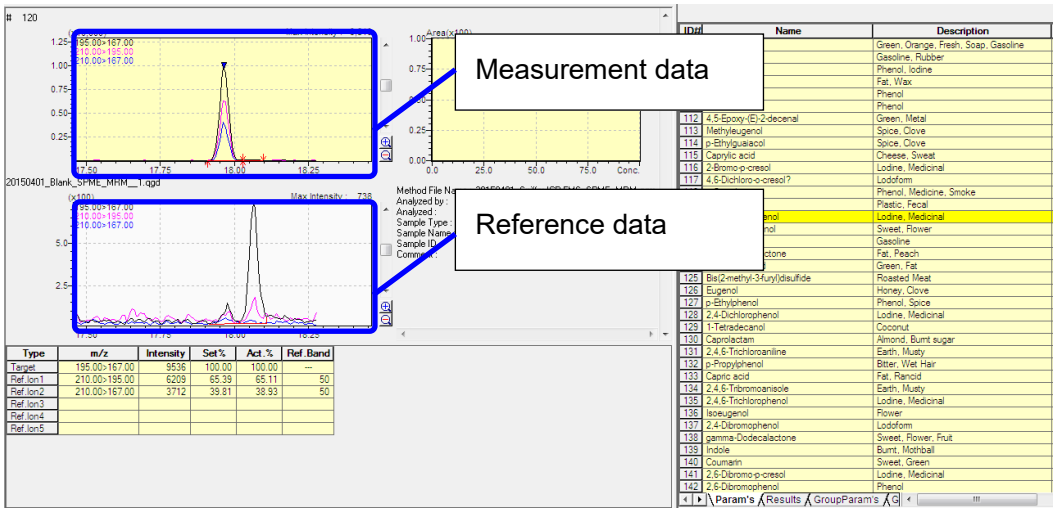
2) Open the data for the sample being examined.

3) Click [Open Reference Data File] on the [File] menu.



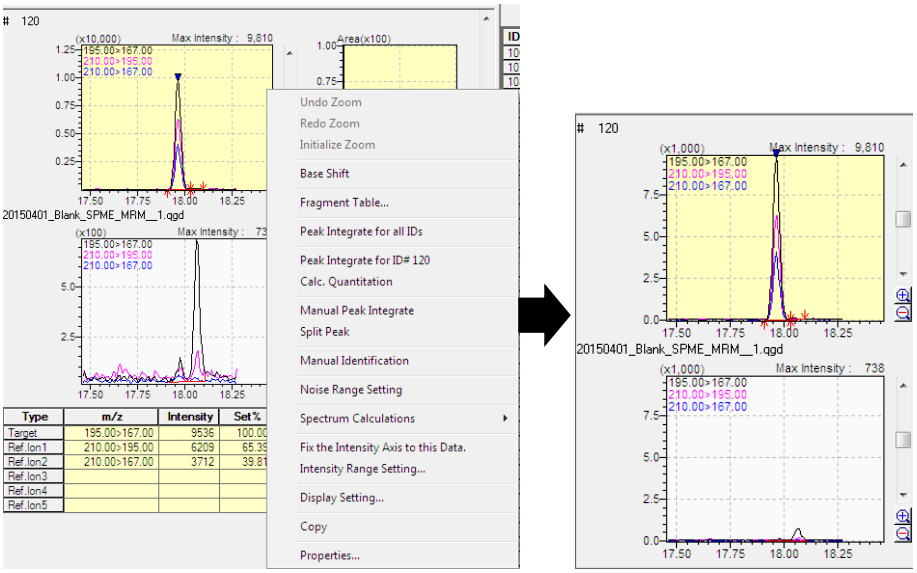
4) Select the data to compare and click [Open].

5) In the ID Chromatogram View, chromatograms of each component in a blank sample and the sample being examined are displayed in the stack mode. Difference in detected components can be compared in multiple sets of data.



Hint

Data can also be compared based on the scale of the intensity axis. Right-click on the quantitative chromatogram for data specified on the intensity axis and click [Fix the Intensity Axis to this Data.] on the right-click menu.



Hint

The information in the compound table can be copied and pasted into Excel. This makes it easy to predict the substances causing an odor by comparing quantitative values for the test samples (odor samples) and blanks (normal samples), and by comparing odor threshold values with the quantitative values of the odor samples.

	A	B	C	D	E	F	G	H
1	ID.	Compound	Odor Quality	Odor Threshold (pg/mg)	Quantitative value(pg/mg)		Ratio(-)	
2					Normal	Abnormal	Abnormal/Odor Threshold	Abnormal/Normal
3	89	Naphthalene	Tar	10.000	0.092	0.084	0.0	0.91
4	90	2-Methylisoborneol	Earth, Musty	0.100	0.017	0.2	2.0	11.76
5	91	Methyl salicylate	Peppermint	1.000	0.009	0.01	0.0	1.11
6	92	alpha-Terpineol	Mint, Anise, Oil	100.000	0.022	0.021	0.0	0.95
7	93	p-Dibromobenzene	Xyene	100.000	0.004	0.004	0.0	1.00
8	94	n-Decanal	Soap, Tallow, Orange peel	1.000	8.479	6.083	6.1	0.72

2.9 Checking the Off-Flavor of Detected Components

For systems that include a sniffer, check the odor by actually smelling the odor of components detected in "*2.8 Analyzing Measurement Data*". The elution times of components that require checking the odor are displayed in advance by the software, which makes it unnecessary to keep sniffing for odors. Instead, the odor can be checked only near the specified time.

2.9.1 Compound Display Settings for the Analysis Sub-Window

This section describes settings for displaying elution times on chromatograms during measurements for components detected in "*2.8 Analyzing Measurement Data*".

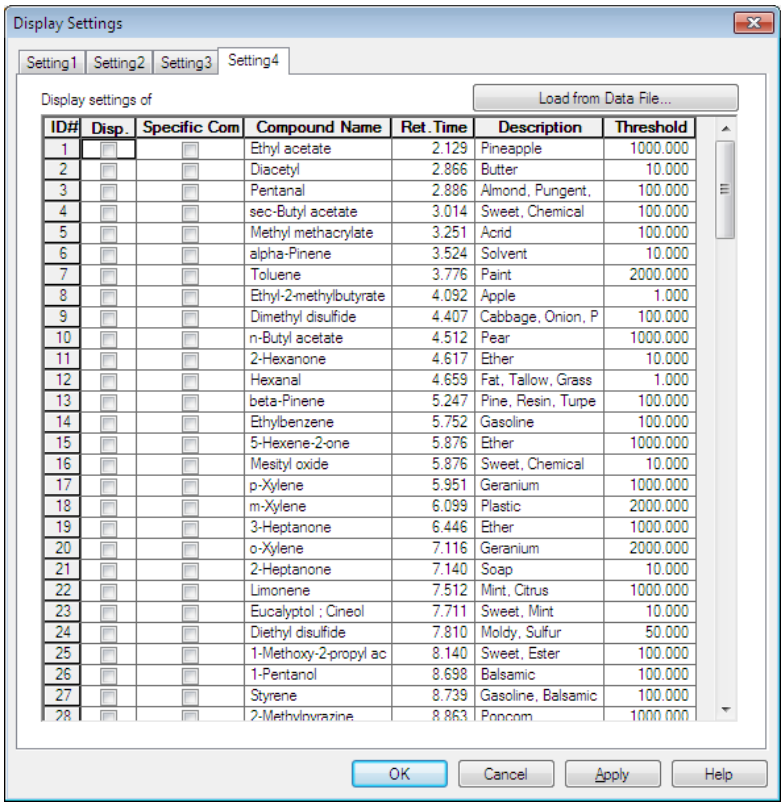
1) Run the [GCMS Real Time Analysis] program.

2) Open the method file for sample analysis that was used in "*2.7 Analyzing Samples*".

3) Click  (Setup Graph) on toolbar.

4) Click the [Setting 4] tab.

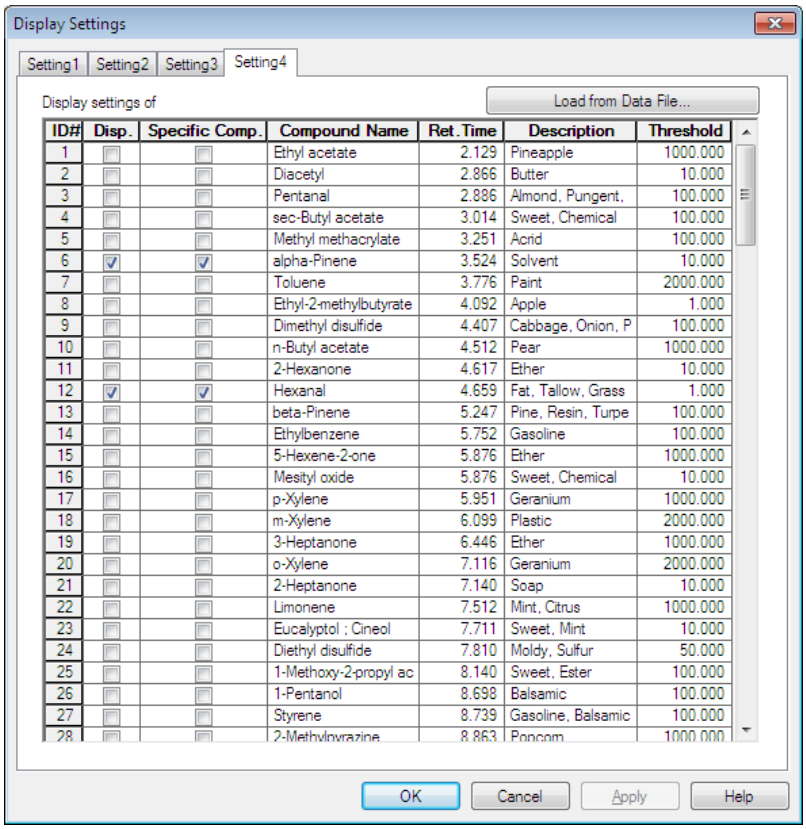
5) Click [Display only identified compounds].



ID#	Disp.	Specific Com	Compound Name	Ret.Time	Description	Threshold
1			Ethyl acetate	2.129	Pineapple	1000.000
2			Diacetyl	2.866	Butter	10.000
3			Pentanal	2.886	Almond, Pungent,	100.000
4			sec-Butyl acetate	3.014	Sweet, Chemical	100.000
5			Methyl methacrylate	3.251	Acrid	100.000
6			alpha-Pinene	3.524	Solvent	10.000
7			Toluene	3.776	Paint	2000.000
8			Ethyl-2-methylbutyrate	4.092	Apple	1.000
9			Dimethyl disulfide	4.407	Cabbage, Onion, P	100.000
10			n-Butyl acetate	4.512	Pear	1000.000
11			2-Hexanone	4.617	Ether	10.000
12			Hexanal	4.659	Fat, Tallow, Grass	1.000
13			beta-Pinene	5.247	Pine, Resin, Turpe	100.000
14			Ethylbenzene	5.752	Gasoline	100.000
15			5-Hexene-2-one	5.876	Ether	1000.000
16			Mesityl oxide	5.876	Sweet, Chemical	10.000
17			p-Xylene	5.951	Geranium	1000.000
18			m-Xylene	6.099	Plastic	2000.000
19			3-Heptanone	6.446	Ether	1000.000
20			o-Xylene	7.116	Geranium	2000.000
21			2-Heptanone	7.140	Soap	10.000
22			Limonene	7.512	Mint, Citrus	1000.000
23			Eucalyptol ; Cineol	7.711	Sweet, Mint	10.000
24			Diethyl disulfide	7.810	Moldy, Sulfur	50.000
25			1-Methoxy-2-propyl ac	8.140	Sweet, Ester	100.000
26			1-Pentanol	8.698	Balsamic	100.000
27			Styrene	8.739	Gasoline, Balsamic	100.000
28			2-Methylpyrazine	8.863	Popcorn	1000.000

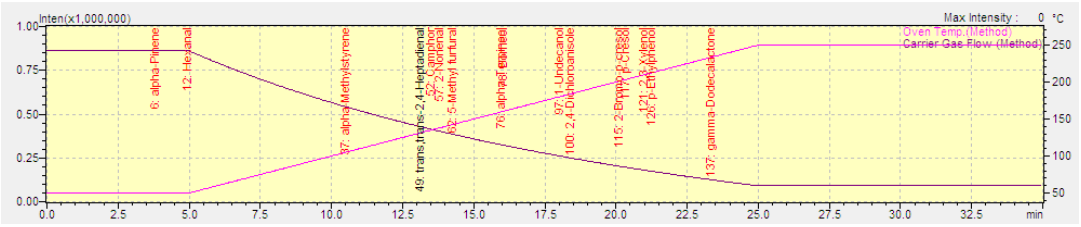
6) Specify the data analyzed in "*2.8 Analyzing Measurement Data*".

7) For identified compounds with component concentrations higher than one thenth of the odor threshold value, the corresponding checkboxes in the [Disp.] column are selected. For concentrations more than ten times the threshold value, the corresponding checkboxes in the [Specific Comp.] column are selected.



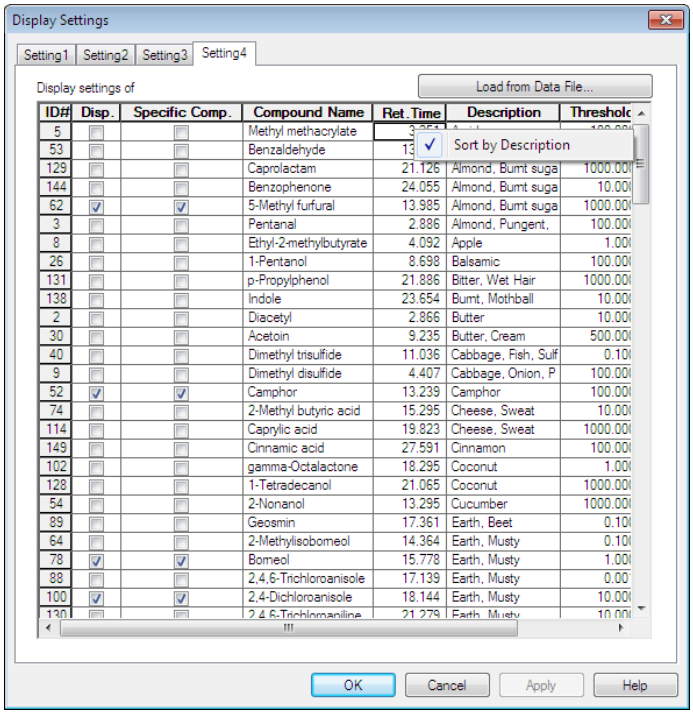
ID#	Disp.	Specific Comp.	Compound Name	Ret.Time	Description	Threshold
1	☐	☐	Ethyl acetate	2.129	Pineapple	1000.000
2	☐	☐	Diacetyl	2.866	Butter	10.000
3	☐	☐	Pentanal	2.886	Almond, Pungent,	100.000
4	☐	☐	sec-Butyl acetate	3.014	Sweet, Chemical	100.000
5	☐	☐	Methyl methacrylate	3.251	Acrid	100.000
6	☑	☑	alpha-Pinene	3.524	Solvent	10.000
7	☐	☐	Toluene	3.776	Paint	2000.000
8	☐	☐	Ethyl-2-methylbutyrate	4.092	Apple	1.000
9	☐	☐	Dimethyl disulfide	4.407	Cabbage, Onion, P	100.000
10	☐	☐	n-Butyl acetate	4.512	Pear	1000.000
11	☐	☐	2-Hexanone	4.617	Ether	10.000
12	☑	☑	Hexanal	4.659	Fat, Tallow, Grass	1.000
13	☐	☐	beta-Pinene	5.247	Pine, Resin, Turpe	100.000
14	☐	☐	Ethylbenzene	5.752	Gasoline	100.000
15	☐	☐	5-Hexene-2-one	5.876	Ether	1000.000
16	☐	☐	Mesityl oxide	5.876	Sweet, Chemical	10.000
17	☐	☐	p-Xylene	5.951	Geranium	1000.000
18	☐	☐	m-Xylene	6.099	Plastic	2000.000
19	☐	☐	3-Heptanone	6.446	Ether	1000.000
20	☐	☐	o-Xylene	7.116	Geranium	2000.000
21	☐	☐	2-Heptanone	7.140	Soap	10.000
22	☐	☐	Limonene	7.512	Mint, Citrus	1000.000
23	☐	☐	Eucalyptol ; Cineol	7.711	Sweet, Mint	10.000
24	☐	☐	Diethyl disulfide	7.810	Moldy, Sulfur	50.000
25	☐	☐	1-Methoxy-2-propyl ac	8.140	Sweet, Ester	100.000
26	☐	☐	1-Pentanol	8.698	Balsamic	100.000
27	☐	☐	Styrene	8.739	Gasoline, Balsamic	100.000
28	☐	☐	2-Methylpyrazine	8.863	Popcorn	1000.000

Confirm that compound names are displayed on the chromatogram at the corresponding elution times for the specified compounds. Compounds specified as a specified substance are indicated in red.



Hint

[Sort by Description] can be selected for sensory information. To undo sorting, click [Sort] again in the [Comment] column.



ID#	Disp.	Specific Comp.	Compound Name	Ret. Time	Description	Threshold
5	☐	☐	Methyl methacrylate			
53	☐	☐	Benzaldehyde	13		
129	☐	☐	Caprolactam	21.126	Almond, Burnt suga	1000.00
144	☐	☐	Benzophenone	24.055	Almond, Burnt suga	10.00
62	☑	☑	5-Methyl furfural	13.985	Almond, Burnt suga	1000.00
3	☐	☐	Pentanal	2.886	Almond, Pungent,	100.00
8	☐	☐	Ethyl-2-methylbutyrate	4.092	Apple	1.00
26	☐	☐	1-Pentanol	8.698	Balsamic	100.00
131	☐	☐	p-Propylphenol	21.886	Bitter, Wet Hair	1000.00
138	☐	☐	Indole	23.654	Burnt, Mothball	10.00
2	☐	☐	Diacetyl	2.866	Butter	10.00
30	☐	☐	Acetoin	9.235	Butter, Cream	500.00
40	☐	☐	Dimethyl trisulfide	11.036	Cabbage, Fish, Sulf	0.10
9	☐	☐	Dimethyl disulfide	4.407	Cabbage, Onion, P	100.00
52	☑	☑	Camphor	13.239	Camphor	100.00
74	☐	☐	2-Methyl butyric acid	15.295	Cheese, Sweat	10.00
114	☐	☐	Caprylic acid	19.823	Cheese, Sweat	1000.00
149	☐	☐	Cinnamic acid	27.591	Cinnamon	100.00
102	☐	☐	gamma-Octalactone	18.295	Coconut	1.00
128	☐	☐	1-Tetradecanol	21.065	Coconut	1000.00
54	☐	☐	2-Nonanol	13.295	Cucumber	1000.00
89	☐	☐	Geosmin	17.361	Earth, Beet	0.10
64	☐	☐	2-Methylisoborneol	14.364	Earth, Musty	0.10
78	☑	☑	Borneol	15.778	Earth, Musty	1.00
88	☐	☐	2,4,6-Trichloroanisole	17.139	Earth, Musty	0.00
100	☑	☑	2,4-Dichloroanisole	18.144	Earth, Musty	10.00
130	☐	☐	2,4,6-Trichloroaniline	21.279	Earth, Musty	10.00

8) Save the method file.

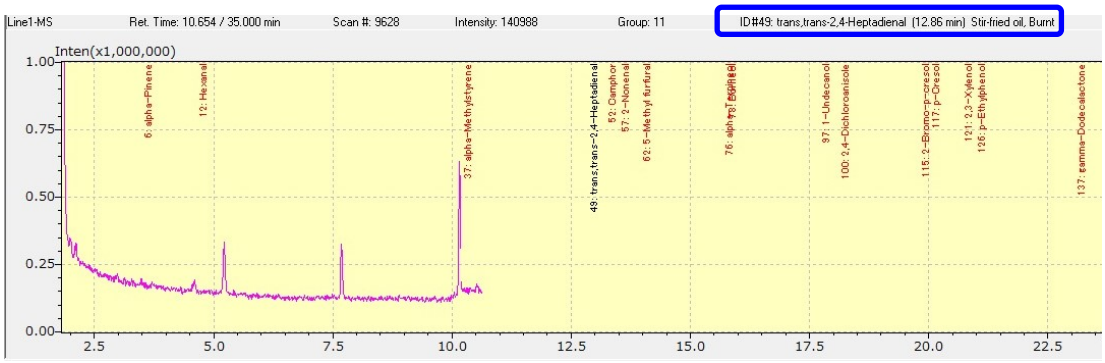
Hint

Display settings are saved in the method file.

2.9.2 Confirming Off-Flavor

1) Analyze the sample using the method file created in "*2.9.1 Compound Display Settings for the Analysis Sub-Window*".

2) Confirm odors near the time the corresponding compound name is displayed on the chromatogram during analysis. Information about the next compound to be eluted (name, retention time, and sensory information) is also available for reference, displayed in the location indicated below.



Hint

The [Display Settings] sub-window can also be kept displayed during analysis. That allows you to view a list of multiple compounds that will be eluted next.

Hint

If multiple compound names displayed on the chromatogram overlap during analysis, they can be enlarged by dragging.Even if the names are displayed enlarged, they automatically scroll as the analysis proceeds.

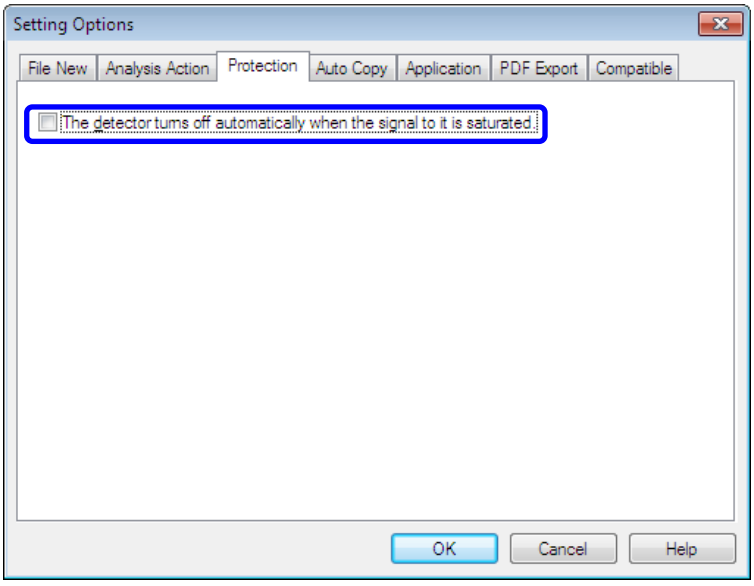
3 Caution

3.1 Changing Analytical Condition and Using an Unspecified Column

The Smart Environmental Database contains retention index obtained under the conditions described in *"Appendix 1 Analytical Conditions."* Be aware that the use of changing analytical condition and/or an unspecified column will entail different retention index, and measurement may not be performed normally.

3.2 Detector Protective Function

Although the detector voltage is preset, some samples may saturate the detector. If the detector has become saturated, the [The detector turns off automatically when the signal to it is saturated.] function is activated, and the filament is turned OFF.
Data cannot be obtained normally when this happens, so be sure to set the detector protective function to OFF.



NOTE

Note that there is a risk that turning OFF the detector protection function can cause a deterioration of the detector. Be sure to fully consider this point before using this function.

Appendix 1. Analytical Conditions

1. MS Parameters (Same for All Methods)

Ion Source Temp	: 200 °C
Interface Temp	: 250 °C

2. GC Parameters

1) InertCap 5MS/Sil (30 m x 0.32 mm I.D.、df = 0.5 μm) Column

Injection Temp	: 250 °C
Column Oven Temp.	: 50 °C (5 min) – (10 °C /min) – 250 °C (10 min)
Purge Flow	: 3mL /min

If a Sniffer Is Not Used

Injection Mode	: Split
Flow Control Mode	: Pressure
Pressure	: 44.5 kPa
Split ratio	: 5

If a Sniffer Is Used

Injection Mode	: Split
Flow Control Mode	: Pressure
Pressure	: 90.0 kPa
Total Flow	: 17.2 mL/min
Split ratio	: -1
Pressure (APC1)	: 20 kPa
Pressure (APC2)	: 200 kPa

2) InertCap 17MS (30 m x 0.25 mm I.D.、df = 0.25 μm) Column

Injection Temp.	: 250 °C
Column Oven Temp.	: 50 °C (5 min) – (10 °C /min) – 250 °C (10 min)
Purge Flow	: 3mL /min

If a Sniffer Is Not Used

Injection Mode	: Split
Flow Control Mode	: Pressure
Pressure	: 83.5 kPa
Split ratio	: 5

If a Sniffer Is Used

Injection Mode	: Split
Flow Control Mode	: Pressure
Pressure	: 160.0 kPa
Total Flow	: 16.4 mL/min
Split ratio	: -1
Pressure (APC1)	: 20 kPa
Pressure (APC2)	: 200 kPa

3) InertCap Pure-Wax (30 m x 0.25 mmI.D.、df = 0.25 μm) Column

Injection Temp.	: 250 °C
Column Oven Temp.	: 50 °C (5 min) – (10 °C /min) – 250 °C (10 min)
Purge Flow	: 3mL /min

If a Sniffer Is Not Used

Injection Mode	: Split
Flow Control Mode	: Pressure
Presure	: 83.5 kPa
Split ratio	: 5

If a Sniffer Is Used

Injection Mode	: Split
Flow Control Mode	: Pressure
Pressure	: 160.0 kPa
Total Flow	: 16.4 mL/min
Split ratio	: -1
Pressure (APC1)	: 20 kPa
Pressure (APC2)	: 200 kPa

3. AOC-20 Parameters

# of Rinses with Solvent (Pre-run)	: 3
# of Rinses with Solvent (Post-run)	: 3
# of Rinses with Sample	: 1
Plunger Speed (Suction)	: High
Viscosity Comp. Time	: 0.2 sec
Plunger Speed (Injection)	: High
Syringe Insertion Speed	: High
Injection Mode	: 0:Normal
Pumping Times	: 5

Inj. Port Dwell Time : 0.3 sec
Plunger Rinsing Speed : High
Rinse Injection Volume : 6 µL

4. AOC-5000 Parameters

1) AOC-5000 Plus (Liquid injection)

Air Volume : 0
Pre Clean with Solvent 1 : 3
Pre Clean with Solvent 2 : 0
Pre Clean with Sample : 2
Filling Volume : 5 µL
Filling Speed : 7 µL/s
Filling Strokes : 5
Pullup Delay : 500 ms
Injection Speed : 10 µL/s
Pre Inject Delay : 0 ms
Post Inject Delay : 0 ms
Post Clean with Solvent 1 : 3
Post Clean with Solvent 2 : 0

2) AOC-5000 Plus (HS)

Incubation Temperature : 60 °C
Incubation Time : 1800 s
Syringe Temperature : 80 °C
Agitator Speed : 250 rpm
Fill Speed : 500 µL/s
Pullup Delay : 500 ms
Injection Speed : 500 µL/s
Pre Inject Delay : 500 ms
Post Inject Delay : 500 ms
Flush Time : 300 s
GC Runtime : Specify a time sufficient to include GC program time, cooling time, and equilibration time, plus an extra margin. If the analysis time is dominated by the pretreatment unit, such as an OPTIC-4 unit, then specify a time sufficient to include pretreatment unit analysis time, cooling time, and equilibration time, plus an extra margin.

3) AOC-5000 Plus (SPME)

Pre Incubation Time	: 300 s
Incubation Temp.	: 80 °C
Pre Inc Agitator Speed	: 250 rpm
Agitator On Time	: 5 sec
Agitator Off Time	: 2 sec
Needle Penetration	: 8 mm
Fiber Penetration in mm	: 14 μL
Extraction Time	: 1800 s
Injection Penetration	: 40 mm
Desorption Time	: 120 s
Post FibCondTime	: 300 s
GC Runtime	: Specify a time sufficient to include GC program time, cooling time, and equilibration time, plus an extra margin. If the analysis time is dominated by the pretreatment unit, such as an OPTIC-4 unit, then specify a time sufficient to include pretreatment unit analysis time, cooling time, and equilibration time, plus an extra margin.

5. AOC-6000 Parameters

1) Liquid Injection

Analysis Group

Syringe Tool	: Select one from the list of syringe tools for injection connected to the instrument (e.g. LS1, LS2).
Pre Wash Cycles	: 3
Sample Rinse Cycles	: 1
Sample Aspirate Flow Rate	: 1 uL/s
Sample Post Aspirate Delay	: 2 s
Injector	: Select one from the list of the injectors connected to the instrument (e.g. Injector1, Injector2).
Injection Flow Rate	: 100 uL/min
Post Wash Cycles	: 3

Setup Group

Gas Chromatograph	: Select one from the list of GC units connected to the instrument (e.g. GC1).

Cooled Stack 1	: (Do not select.)
Cooled Stack 1 Temperature	: 20 °C
Cooled Stack 2	: (Do not select.)
Cooled Stack 2 Temperature	: 20 °C
Pre Wash Station	: Select one from the list of wash stations connected to the instrument.
Post Wash Station	: Select one from the list of wash stations connected to the instrument.
Wash Vial Depth	: 40 mm
Waste Port Depth	: 10 mm
Sample Vial Depth	: 30 mm
Bottom Sense Sample Vial	: Off
Height From Bottom Sample Vial	: 3 mm
Injection Mode	: Normal
Injection Signal Mode	: PlungerDown
Injector Penetration Depth	: 40 mm
Pre Ahead Option	: Disabled

Advanced Group

Pre Wash Solvent Volume	: 8 uL
Pre Wash Solvent Position Step 1	: 1
Pre Wash Solvent Position Step 2	: 0
Pre Wash Solvent Position Step 3	: 0
Pre Wash Solvent Position Step 4	: 0
Pre Wash Aspirate Flow Rate	: 5 uL/s
Sample Vial Penetration Speed	: 50 mm/s
Sample Rinse Volume	: 6 uL
Filling Strokes Count	: 5
Filling Strokes Volume	: 6 uL
Filling Strokes Aspirate Flow Rate	: 5 uL/s
Filling Strokes Post Aspirate Delay	: 0.2 s
Filling Strokes Post Dispense Delay	: 0.5 s
Delay After Filling Strokes	: 0.5 s
Air Gap Volume	: 0 uL
Injector Penetration Speed	: 100 mm/s
Pre Injection Dwell Time	: 0 s
Post Injection Dwell Time	: 0.3 s

Post Wash Solvent Volume	: 8 uL
Post Wash Solvent Position Step 1	: 1
Post Wash Solvent Position Step 2	: 0
Post Wash Solvent Position Step 3	: 0
Post Wash Solvent Position Step 4	: 0
Post Wash Aspirate Flow Rate	: 5 uL/s

2) Headspace Injection

Analysis Group

Syringe Tool	: Select one from the list of syringe tools for HS connected to the instrument (e.g. HS1, HS2).
Incubation Temperature	: 60 °C
Incubation Time	: 30 min
Syringe Temperature	: 80 °C
Agitator Speed	: 250 rpm
Pre Purge Time	: 5 s
Injector	: Select one from the list of the injectors connected to the instrument (e.g. Injector1, Injector2).
Injection Flow Rate	: 30 mL/min
Post Purge Time	: 10 s
Analysis Time	: Specify a time sufficient to include GC program time, cooling time, and equilibration time, plus an extra margin. If the analysis time is dominated by the pretreatment unit, such as an OPTIC-4 unit, then specify a time sufficient to include pretreatment unit analysis time, cooling time, and equilibration time, plus an extra margin.

Setup Group

Gas Chromatograph	: Select one from the list of GC units connected to the instrument (e.g. GC1).
Sync Before Incubation End Time	: 0 min
Agitator	: Select one from the list of trays connected to the instrument.
Heat Agitator	: TRUE
Wait For Readiness Agitator	: TRUE
Sample Vial Depth	: 15 mm

Heat Syringe	: TRUE
Wait For Readiness Syringe	: TRUE
Injection Signal Mode	: PlungerUp
Injector Penetration Depth	: 40 mm
Continuous Purge	: FALSE

Advanced Group

Filling Strokes Count	: 0
Filling Strokes Volume	: 2 mL
Filling Strokes Aspirate Flow Rate	: 30 mL/min
Delay After Filling Strokes	: 0.5 s
Sample Aspirate Flow Rate	: 30 mL/min
Sample Post Aspirate Delay	: 0.5 s
Sample Vial Penetration Speed	: 50 mm/s
Injector Penetration Speed	: 50 mm/s
Pre Injection Dwell Time	: 0.5 s
Post Injection Dwell Time	: 0.5 s
Agitator On Time	: 5 s
Agitator Off Time	: 2 s

3) SPME Injection

Analysis Group

Fiber Tool	: Select one from the list of syringe tools for SPME connected to the instrument.
Conditioning Port	: Select one from the pull-down list.
Conditioning Temperature	: 270 °C
Pre Conditioning Time	: 0 min
Incubation Temperature	: 80 °C
Incubation Time	: 5 min
Agitator Speed	: 250 rpm
Sample Vial Depth	: 22 mm
Sample Extract Time	: 30 min
Injector	: Select one from the list of the injectors connected to the instrument (e.g. Injector1, Injector2).
Sample Desorb Time	: 2 min
Post Conditioning Time	: 5 min
Analysis Time	: Specify a zero-time that includes the GC program

time, cooling time, and equilibration time, plus an extra margin. If the analysis time is dominated by the pretreatment unit, such as an OPTIC-4 unit, then specify a time sufficient to include pretreatment unit analysis time, cooling time, and equilibration time, plus an extra margin.

Setup Group

Gas Chromatograph	: Select one from the list of GC units connected to the instrument (e.g. GC1).
Sync Before Extraction End Time	: 0 min
Agitator	: Select one from the list of trays connected to the instrument.
Do Agitation	: TRUE
Heat Agitator	: TRUE
Wait For Readiness Agitator	: TRUE
Internal Standard Station	:(Do not select.)
Internal Standard Position	: 1
Injector Penetration Depth	: 54 mm
Injection Signal Mode	: Before Expose

Advanced Group

Internal Standard Adsorb Time	: 0 min
Internal Standard Penetration Depth	: 32 mm
Sample Vial Penetration Speed	: 20 mm/s
Injector Penetration Speed	: 100 mm/s
Agitator On Time	: 5 s
Agitator Off Time	: 2 s

6. OPTIC-4 Parameters

1) InertCap 5MS/Sil (30 m x 0.32 mm I.D., df = 0.5 μm) Column

Equilibration Time	: 5 sec.
End Time	: 57 min.
Initial Temp.	: 40 °C
Delay Time	: 0 sec

Ramp Rate1	: 50 °C /sec
Hold Temp.	: 250 °C
Solvent Cooling Effect	: No
Cooling Valve Mode	: No
Septum Purge Flow	: 3 mL/min
Vent Mode	: Fixed Time
Vent Time	: 0 sec
Carrier Control Mode	: Pressure
Zero LINEX Head Pressure	: No

<u>If a Sniffer Is Not Used</u>

Initial Column Flow[*1]	: 1 mL/min
Initial Inlet Pressure	: 44.5 kPa
Start Inlet Pressure1	: 44.5 kPa
End Inlet Pressure1	: 44.5 kPa
Initial Split Flow	: 11.9 mL/min
Split Flow1	: 11.9 mL/min

<u>If a Sniffer Is Used</u>

Initial Column Flow[*1]	: 1 mL/min
Initial Inlet Pressure	: 90.0 kPa
Start Inlet Pressure1	: 90.0 kPa
End Inlet Pressure1	: 90.0 kPa
Initial Split Flow	: 12.2 mL/min
Split Flow1	: 12.2 mL/min

2) InertCap 17MS (30 m x 0.25 mm I.D., df = 0.25 µm) Column

Equilibration Time	: 5 sec.
End Time	: 45 min.
Initial Temp.	: 40 °C
Delay Time	: 0 sec
Ramp Rate1	: 50 °C /sec
Hold Temp.	: 250 °C
Solvent Cooling Effect	: No
Cooling Valve Mode	: No
Septum Purge Flow	: 3 mL/min

Vent Mode	: Fixed Time
Vent Time	: 0 sec
Carrier Control Mode	: Pressure
Zero LINEX Head Pressure	: No

<u>If a Sniffer Is Not Used</u>

Initial Column Flow*1	: 1 mL/min
Initial Inlet Pressure	: 83.5 kPa
Start Inlet Pressure1	: 83.5 kPa
End Inlet Pressure1	: 83.5 kPa
Initial Split Flow	: 7.2 mL/min
Split Flow1	: 7.2 mL/min

<u>If a Sniffer Is Used</u>

Initial Column Flow*1	: 1 mL/min
Initial Inlet Pressure	: 160.0 kPa
Start Inlet Pressure1	: 160.0 kPa
End Inlet Pressure1	: 160.0 kPa
Initial Split Flow	: 11.2 mL/min
Split Flow1	: 11.2 mL/min

*1: Initial column flow setting values are specified as a formality for executing method files. They have no effect on analysis.

3) InertCap Pure-Wax (30 m x 0.25 mmI.D., df = 0.25 μm) Column

Equilibration Time	: 5 sec.
End Time	: 45 min.
Initial Temp.	: 40 °C
Delay Time	: 0 sec
Ramp Rate1	: 50 °C /sec
Hold Temp.	: 250 °C
Solvent Cooling Effect	: No
Cooling Valve Mode	: No
Septum Purge Flow	: 3 mL/min
Vent Mode	: Fixed Time
Vent Time	: 0 sec
Carrier Control Mode	: Pressure

Zero LINEX Head Pressure : No

If a Sniffer Is Not Used

Initial Column Flow*1 : 1 mL/min
Initial Inlet Pressure : 83.5 kPa
Start Inlet Pressure1 : 83.5 kPa
End Inlet Pressure1 : 83.5 kPa
Initial Split Flow : 7.2 mL/min
Split Flow1 : 7.2 mL/min

If a Sniffer Is Used

Initial Column Flow*1 : 1 mL/min
Initial Inlet Pressure : 160.0 kPa
Start Inlet Pressure1 : 160.0 kPa
End Inlet Pressure1 : 160.0 kPa
Initial Split Flow : 11.2 mL/min
Split Flow1 : 11.2 mL/min

*1: Initial column flow setting values are specified as a formality for executing method files. They have no effect on analysis.

Appendix 2. List of Customer-Supplied Items

The following reagents and columns are required for creating method files using the GC/MS odor analysis system. The customer must provide the following reagents and columns, which are not included with the GC/MS odor analysis system.

<Reagent>

Sample for adjusting retention times

- C7 - C30 Saturated Alkanes (1000 µg/mL each component in hexane)	Cat：49451-U SIGMA-ALDRICH

Samples for evaluating instrument performance

- Acetophenone solution (2000 µg/mL in methylene chloride)	Cat: APP-9-004-20X FUJIFILM Wako Pure Chemical
- Naphthalene solution (5000 µg/mL in methanol)	Cat: 40053 SIGMA-ALDRICH
- 2,6-Dichlorophenol solution (5000 µg/mL in methanol)	Cat: APP-9-076-M-50X FUJIFILM Wako Pure Chemical
- 2,4,6-Trichloroanisole solution (100/mL in methanol)	Cat: 47526-U SIGMA-ALDRICH

Samples for correcting calibration curves

- Internal Standards / Surrogate Standards Mix EPA524.1、EPA524.2 Cat: 47358-U
  (2000 µg/mL 4-Bromofluorobenzene, 1,2-Dichlorobenzene-d4, Fluorobenzene in methanol) SIGMA-ALDRICH
- Acenaphthene-d10 solution Cat: 48417
  (2000 µg/mL component in methylene dichloride) SIGMA-ALDRICH

<Columns>

Low-polarity column
InertCap 5MS/Sil (30 m x 0.32 mm I.D., df = 0.5 µm) P/N: 1010-15244
Mid-polarity column
InertCap 17MS (30 m x 0.25 mm I.D., df = 0.25 µm) P/N: 1010-2014
High-polarity column
InertCap Pure-Wax (30 m x 0.25 mm I.D., df = 0.25 µm) P/N: 1010-68142
GL Science Inc.

For information about consumables used in respective pretreatment units, refer to the instruction manual for the applicable pretreatment unit.

Appendix 3. Standard Sample Preparation Method

The following describes how to prepare respective standard samples. The following equipment, supplies, and organic solvents, are required for preparing standard samples.

<Equipment>

- 10mL volumetric flask (5mL is acceptable)
- Microsyringe
- Storage vials

<Organic Solvents>

- Hexane
- Methanol

Appendix 3.1. Preparing Standard Samples for Adjusting Retention Times

The following standard samples are recommended for use as standard samples for adjusting retention times.

- C7 - C30 Saturated Alkanes (1000 µg/mL each component in hexane) Cat: 49451-U Supplier:SIGMA-ALDRICH

Standard sample for adjusting retention time (50 µg/mL of each alkane)

1) Measure 500 µL of C7 to C30 saturated alkanes with a microsyringe and add them to a 10 mL volumetric flask.
2) Add hexane to make 10 mL.
3) Transfer mixture to a storage vial and store refrigerated.

Appendix 3.2. Preparing Standard Samples for Evaluating Instrument Perfomance

The following standard samples are recommended for use as standard samples for evaluating I nstrument performance.

- Acetophenone solution (2000 µg/mL in methylene chloride) Cat: APP-9-004-20X FUJIFILM Wako Pure Chemical
- Naphthalene solution (5000 µg/mL in methanol) Cat: 40053 Supplier : SIGMA-ALDRICH
- 2,6-Dichlorophenol solution (5000 µg/mL in methanol) Cat: APP-9-076-M-50X FUJIFILM Wako Pure Chemical
- 2,4,6-Trichloroanisole solution (100 µg/mL in methanol) Cat: 47526-U Supplier : SIGMA-ALDRICH

Standard samples for evaluating instrument performance

- Acetophenone : 2 µg/mL
- Naphthalene : 5 µg/mL
- 2,6-Dichlorophenol : 5 µg/mL
- 2,4,6-Trichloroanisole : 0.1 µg/mL

1) Add about 5mL of methanol to a 10 mL volumetric flask.
2) Measure 10 µLeach of standard samples with microsyringe and add them to a 10 mLvolumetric flask.
3) Add methanol to make 10mL.
4) Transfer mixture to a storage vial and store refrigerated.

Appendix 3.3. Preparing Standard Samples for Correcting Calibration Curves

The following standard samples are recommended for use as standard samples for correcting calibration curves.

- Internal Standards / Surrogate Standards Mix EPA524.1、EPA524.2 Cat: 47358-U
  (2000 µg/mL 4-Bromofluorobenzene, 1,2-Dichlorobenzene-d4, Fluorobenzene in methanol) Supplier : SIGMA-ALDRICH
  *1 Fluorobenzene is included in the standard sample, but is not a target component.
- Acenaphthene-d10 solution Cat: 48417
  (2000 µg/mL component in methylene dichloride) Supplier : SIGMA-ALDRICH

Standard samples for correcting calibration curves

- 4-Bromofluorobenzene : 0.1 µg/mL
- 1,2-Dichlorobenzene-d4 : 0.1 µg/mL
- Acenaphthene-d10l : 0.1 µg/mL

1) Prepare a standard mixture solution (1000 µg/mL) by measuring 500 µL each of an internal standard/surrogate standard substance mixture and acenaphthene-d10 using a microsyringe and adding them to a GC vial.
2) Add about 5 mL of hexane to a 10 mL volumetric flask.
3) Measure 100 µL of the standard mixture sample prepared above with a microsyringe and add it to the 10 mL volumetric flask.
4) Add hexane to make 10 mL.
5) Transfer mixture to a storage vial.
6) Add about 5 mL of hexane to a 10 mL volumetric flask. Use a volumetric flask that is

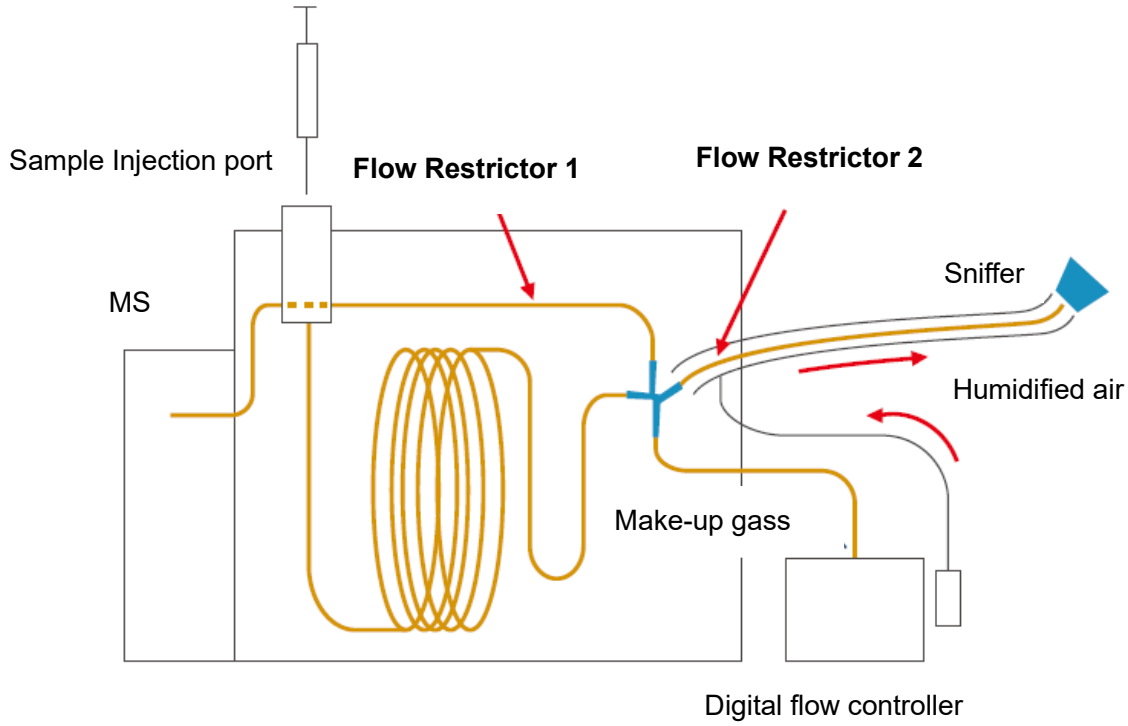
different from the one used in Step 2).

7) Measure 100 μL of the standard mixture sample prepared in Step 5) with a microsyringe and add it to the 10 mL volumetric flask.
8) Add hexane to make 10 mL.
9) Transfer mixture to a storage vial and store refrigerated.

Appendix 4. Flow Restrictor Setting for Connecting the Sniffer

If connecting a sniffer to the GC/MS system, use a flow restrictor with a 4-way valve installed and a digital flow controller (APC) to maintain a constant split ratio for splitting the flow of components eluted from the column. The split ratio in the GC/MS odor analysis system has been optimized. Therefore, install the following flow restrictor.

	I.D.(mm)	Length(m)	Connected To	Flowrate Split Ratio (MS:Sniffer)
Flow Restrictor 1 (MS end)	0.15	1.7	MS	Approx. 0.5:1
Flow Restrictor 2 (Sniffer unit end)	0.25	2.0	Sniffer	



Appendix 5. Replacing Columns If a Sniffer Is Used

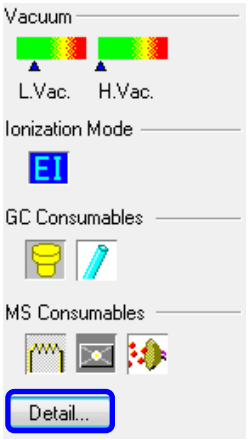
Systems with a sniffer installed allow columns to be replaced without stopping the vacuum in the MS unit. The following describes how to replace columns without stopping the vacuum in the MS unit.

The column replacement procedure differs depending on whether an SPL unit or OPTIC-4 unit is used as the sample injection unit. Replace the column according to the procedure in "*Appendix 5.1 Replacing Columns When using an SPL Injection Unit*" or the procedure in "*Appendix 5.2 Replacing Coluns When Using an OPTIC-4 Inlet*".

Appendix 5.1. Replacing Columns When using an SPL Injection Unit

1) Prepare the method file and folder to be used after the column is replaced according to the procedure in "*2.1 Preparation*".

2) Click [Detail] in the [GCMS Real Time Analysis] program. The [Monitor Settings] sub-window is displayed.



3) Click the [Consumable] tab.

4) Click the 

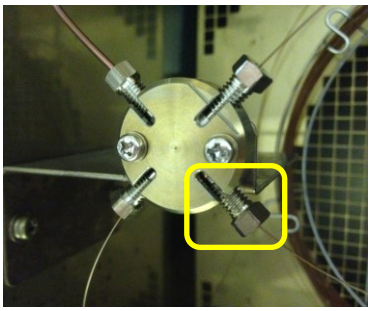
icon.

5) When the status indicates "Ready", click [Replace].

6) Open the column oven door and disconnect the nut at the end of the column from the SPL unit.

7) Unscrew the set screws, installed at the outlet end of the column, from the branch

element. Then remove the column.



NOTE

The position of the set screws actually installed may be different from shown above. Therefore, be sure to confirm that the end of the column is connected before unscrewing the set screws.

NOTE

When the set screws are removed from the branch element, carrier gas delivered from the APC unit will begin leaking out from the branch element. Therefore, once the set screws are removed, replace the column promptly.

8) Install the new column.

9) After installing the column, click [Complete]. After the system automatically checks for air leaking into the system and confirms that the leakage is less than the criterion value, the sample injection unit, column, and interface temperatures return to their previous temperatures before Easy sTop functioned.

10) In the [GCMS Real Time Analysis] program, register the column information in the [System Configuration] sub-window.

11) Load a method file that is compatible with the installed column.

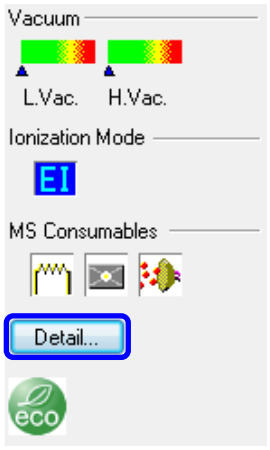
12) Click [Download Initial Parameters] on the [Acquisition] menu.

> **NOTE**
>
> The GC unit controls the flowrate of carrier gas based on the column information specified in configuration settings. If the column specified in configuration settings is different from the column installed in the GC unit, then GC flowrate cannot be controlled properly and an error may be displayed. If the column was replaced, be sure to update the column information in configuration settings promptly.

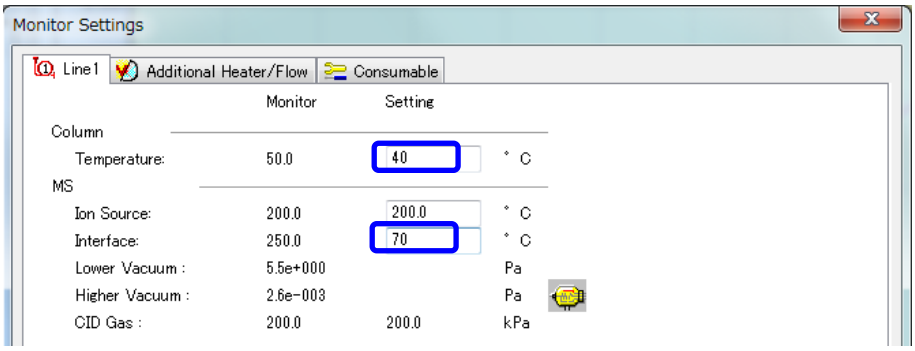
Appendix 5.2. Replacing Coluns When Using an OPTIC-4 Inlet

1) Prepare the method file and folder to be used after the column is replaced according to the procedure in "*2.1 Preparation*".

2) Click [Detail] in the [GCMS Real Time Analysis] program.
The [Monitor Settings] sub-window is displayed.



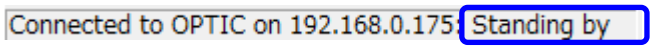
3) Set 70 °C at [Interface Temperature], set 40 °C at [Column Temperature], and click [Apply].



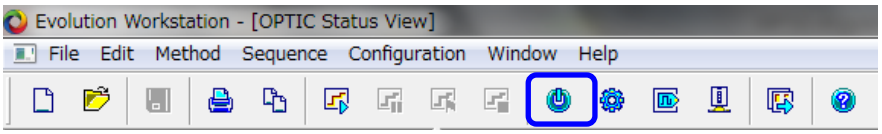
4) Click [OK] to close the sub-window.

5) Wait until Interface temperatures reach 70 °C and the column temperature reaches 40 °C

6) Confirm that the system status indicated in the [OPTIC Status View] sub-window of the Evolution Workstation is "Standing by."

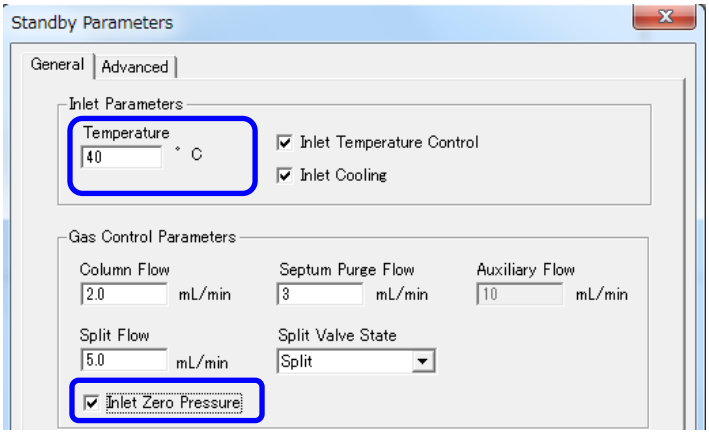


7) Click [Standby Parameters].

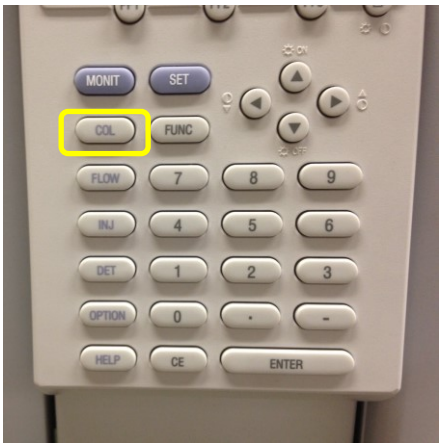


8) Set 40 °C at [Temperature].

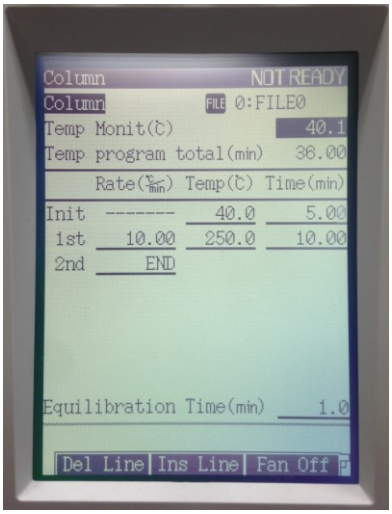
9) Select the [Inlet Zero Pressure] checkbox and click [OK].



10) Press the [COL] button on the GC panel.

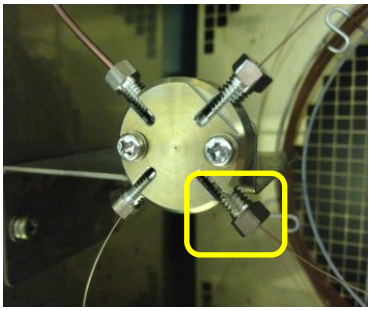


11) Press the [PF3] button to switch [Fan] OFF.



12) Open the column oven door and disconnect the nut at the end of the column from the OPTIC-4 unit.

13) Unscrew the set screws, installed at the end of the column, from the branch element.



NOTE

The actual position of the set screws installed at the end of the column may be different from shown above. Therefore, be sure to confirm that the end of the column is connected before unscrewing the set screws.

NOTE

When the set screws are removed from the branch element, carrier gas delivered from the APC unit will begin leaking out from the branch element. Therefore, once the set screws are removed, replace the column promptly.

14) Install the new column.

15) Press the [COL] button on the GC panel.

16) Press the [PF3] button to switch [Fan] ON.

17) In the [GCMS Real Time Analysis] program, register the column information in the [System Configuration] sub-window.

18) Specify Evolution Workstation configuration settings.
For setting instructions, see "*2.3.3 Using an OPTIC-4 Inlet*".

19) Load a method file that is compatible with the installed column.

20) Click [Download Initial Parameters] on the [Acquisition] menu.

NOTE

The OPTIC-4 unit controls the flowrate of carrier gas based on the column information specified in system configuration settings. If the specified column is different from the column actually installed in the GC unit, then GC flowrate cannot be controlled properly and an error may be displayed. If the column was replaced, be sure to update the column information promptly.

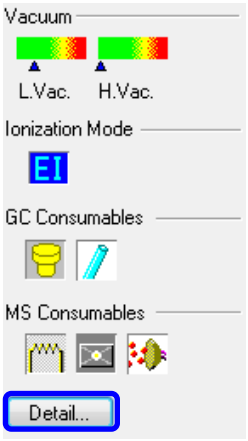
Appendix 6. Changing from an SPL Sample Injection to an OPTIC-4 Inlet

If both an SPL injection unit and OPTIC-4 inlet are installed in the same GC system, then sample injection unit can be changed from the SPL unit to the OPTIC-4 unit without stopping the vacuum in the MS unit. To change from an OPTIC-4 unit to an SPL unit, see "*Appendix 7 Changing from an OPTIC-4 Inlet to an SPL Sample Injection Unit*".

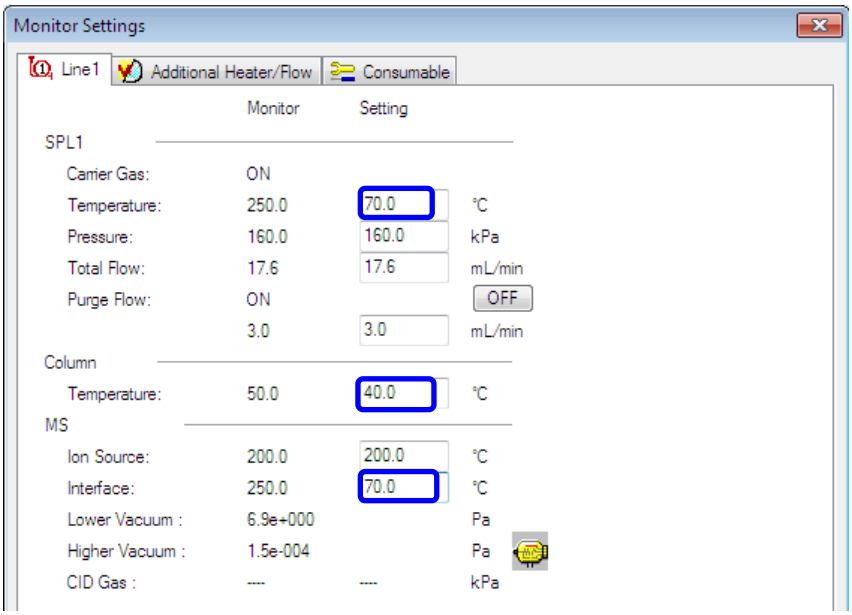
1) Prepare the method file and folder compatible with the column used, according to the procedure in "*2.1 Preparation*"

.

2) Click [Detail] in the [GCMS Real Time Analysis] program The [Monitor Settings] sub-window is displayed.



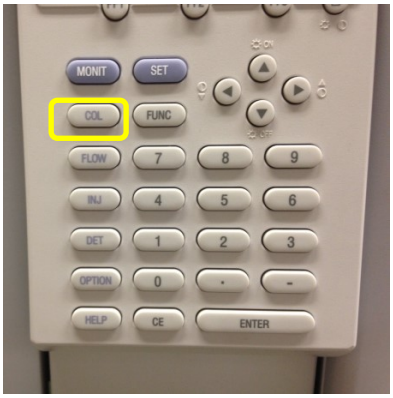
3) Set 70 °C at [Injection Port Temperature] and [Interface Temperature], set 40 °C at [Column Temperature], and click [Apply].



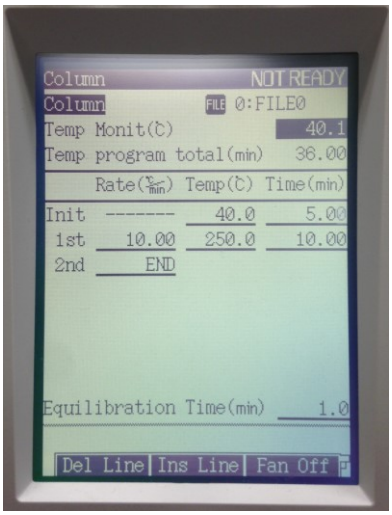
4) Click [OK] to close the sub-window.

5) Wait until the sample injection unit (SPL) and interface temperatures reach 70 °C and the column temperature reaches 40 °C.

6) Press the [COL] button on the GC panel.

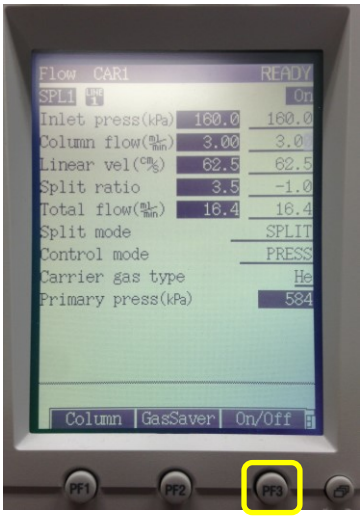


7) Press the [PF3] button to switch [Fan] OFF.



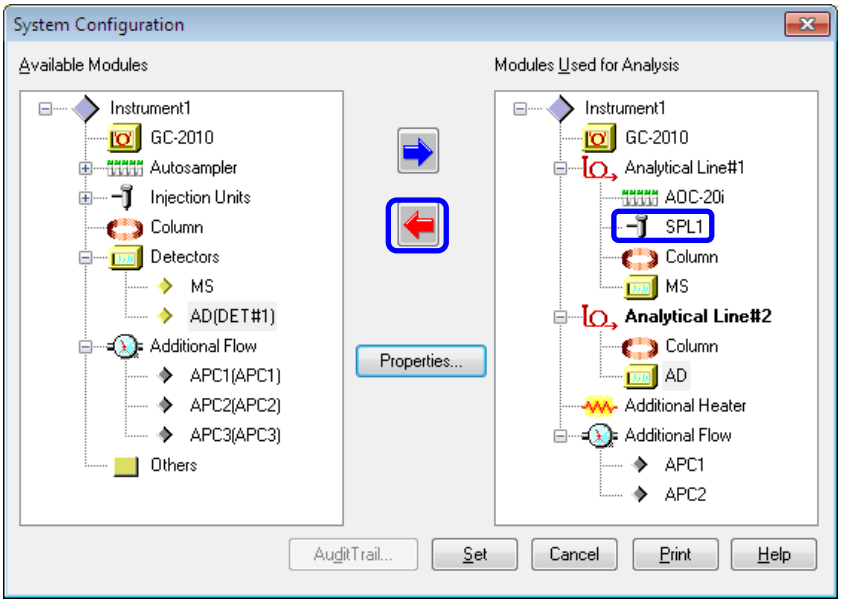
8) Press the [FLOW] button on the GC panel.

9) Press the [PF3] button to switch flowrate control OFF.



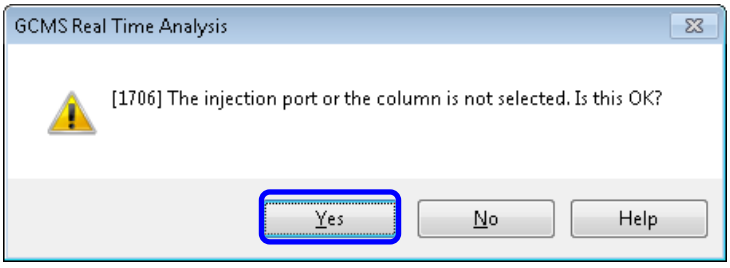
10) Click the [System Configuration] icon on the [Real Time] assistant bar.

11) Select [SPL] at [Analytical Line#1] and remove it from [Modules Used for Analysis].



12) Click [Set].

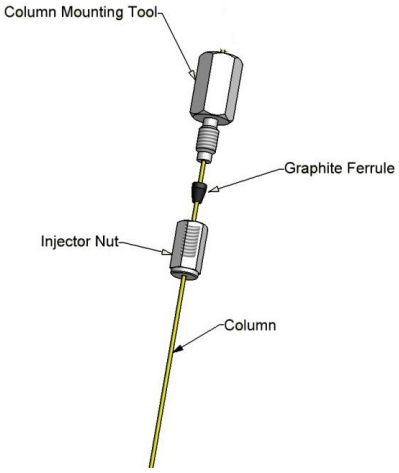
13) When the following sub-window is displayed, click [Yes].



14) Open the column oven door and disconnect the nut at the end of the column from the SPL unit.

15) Remove the nut and ferrule for the SPL unit from the end of the column.

16) Use the nut and ferrule for the OPTIC-4 unit to connect the column to the OPTIC-4 column mounting tool.
Install the ferrule with the tapered end inside the nut. Hand tighten the nut. If the column moves easily, use the provided column nut wrench to tighten it until it cannot be moved.



17) Disconnect the column from the column mounting tool and insert it into the column port on the OPTIC-4 unit. Tighten the nut securely by hand.

18) Use the provided column nut wrench to tighten the nut one half rotation.
When finished connecting the column, inspect the connections for any gas leaks.

NOTE

Be careful not to overtighten the column nut. The column connection on the injection inlet end is extremely narrow and easily damaged.

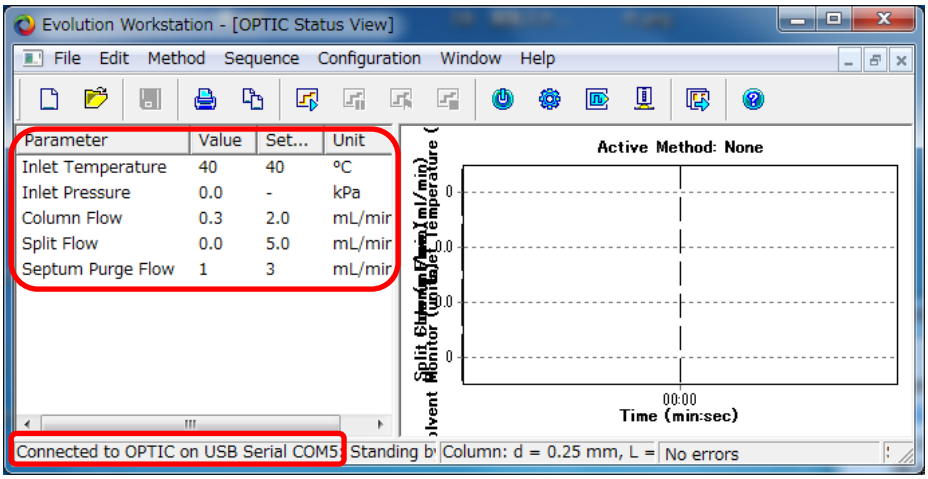
19) Close the column oven door.

20) Press the [COL] button on the GC panel.

21) Press the [PF3] button to switch [Fan] ON.

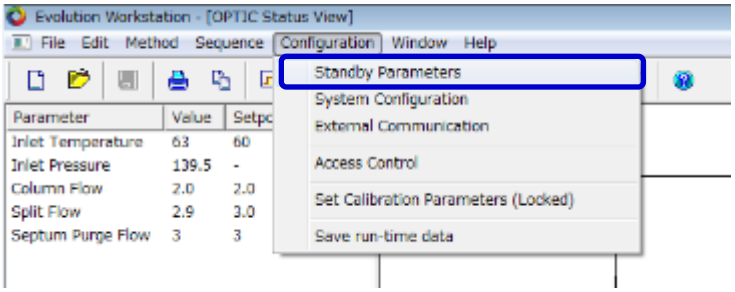
22) Check that the OPTIC-4 is turned ON.

23) Run the Evolution Workstation, and check the status display window.
When communication is established between the computer and OPTIC-4 control unit, the standby status parameter setting values and current values are displayed in the [OPTIC Status View] sub-window. The system status display area indicates the connection method and port number used to connect to the computer.

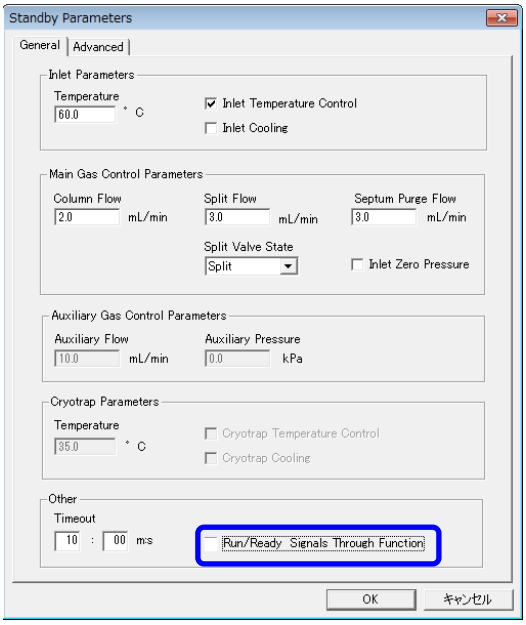


24) Specify configuration settings. For setting instructions, see "*2.3.3 Using an OPTIC-4 Inlet*".

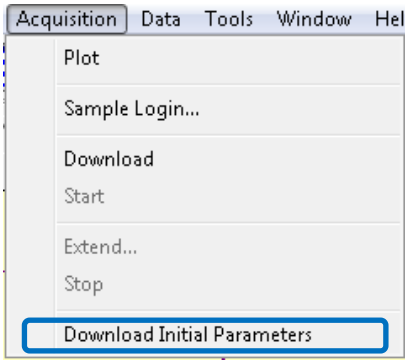
25) In the Evolution Workstation, display the [Standby Parameters] sub-window.



26) In the [Standby Parameters] sub-window, clear the [Run/Ready Signals Through Function] checkbox.



27) Load the method file and click [Download Initial Parameters] on the [Acquisition] menu.



28) When each parameter value reaches its setting value, the status of both the [GC] and [MS] units is [Ready], then the switch from the SPL unit to the OPTIC-4 unit is finished.

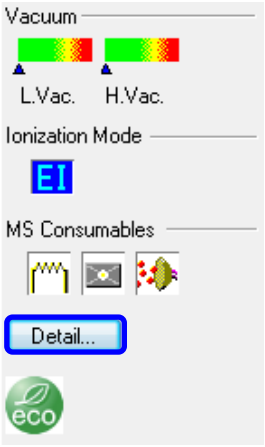
Appendix 7. Changing from an OPTIC-4 Inlet to an SPL Sample Injection Unit

If both an SPL injection unit and OPTIC-4 inlet are installed in the same GC system, then sample injection unit can be changed from the OPTIC-4 unit to the SPL unit without stopping the vacuum in the MS unit. To change from an SPL unit to an OPTIC-4 unit, see Appendix 6 Changing from an SPL Sample Injection Unit to an OPTIC-4 Inlet.

1) Prepare the method file and folder compatible with the column used, according to the procedure in "*2.1 Preparation*"

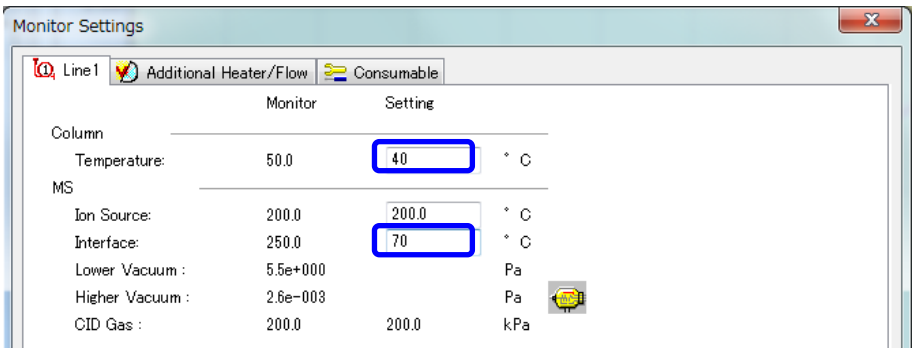
.

2) Click [Detail] in the [GCMS Real Time Analysis] program.



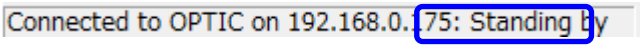
3) Click the [Line 1] tab.

4) Set 70 °C at [Interface Temperature] and 40 °C at [Column Temperature], and click [Apply].

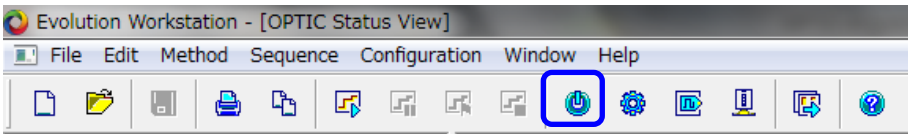


5) Wait until the interface temperature reaches 70 °C and the column temperature reaches 40 °C.

6) Confirm that the system status indicated in the [OPTIC Status View] sub-window of the Evolution Workstation is "Standing by."

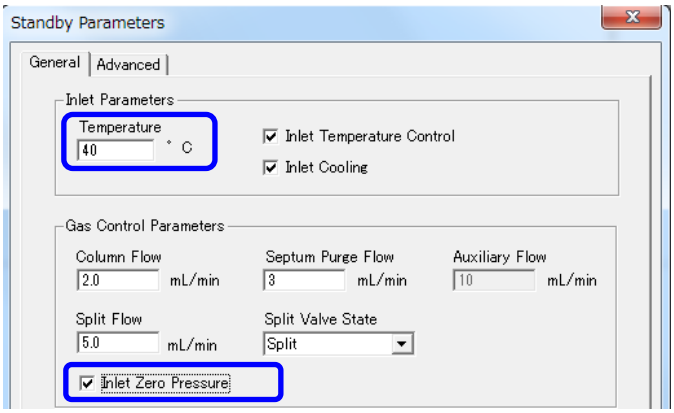


7) Click [Standby Parameters].



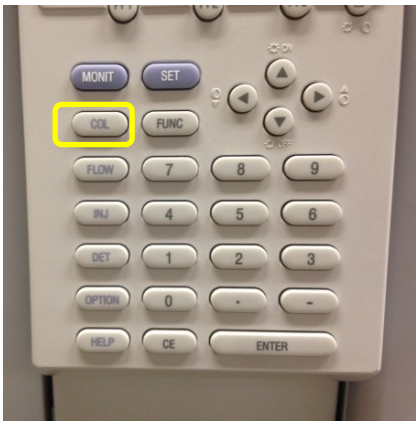
8) Set 40 °C at [Temperature].

9) Select the [Inlet Zero Pressure] checkbox and click [OK].

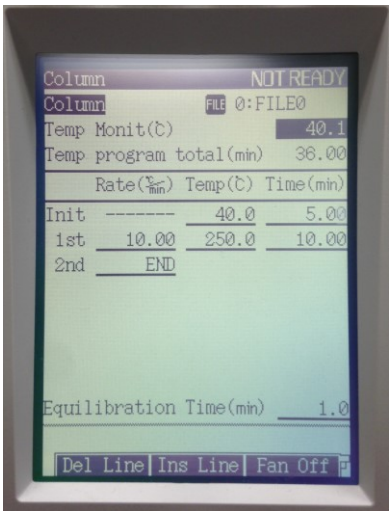


10) Click [OK] to close the sub-window.

11) Press the [COL] button on the GC panel.



12) Press the [PF3] button to switch [Fan] OFF.



13) Open the column oven door and disconnect the nut at the end of the column from the OPTIC-4 unit.

14) Remove the nut and ferrule for the OPTIC-4 unit from the end of the column.

15) Attach the ferrule and nut for the SPL unit to the end of the column.

16) Connect the nut from the end of the column to the end of the SPL unit.

17) Press the [FLOW] button on the GC panel.

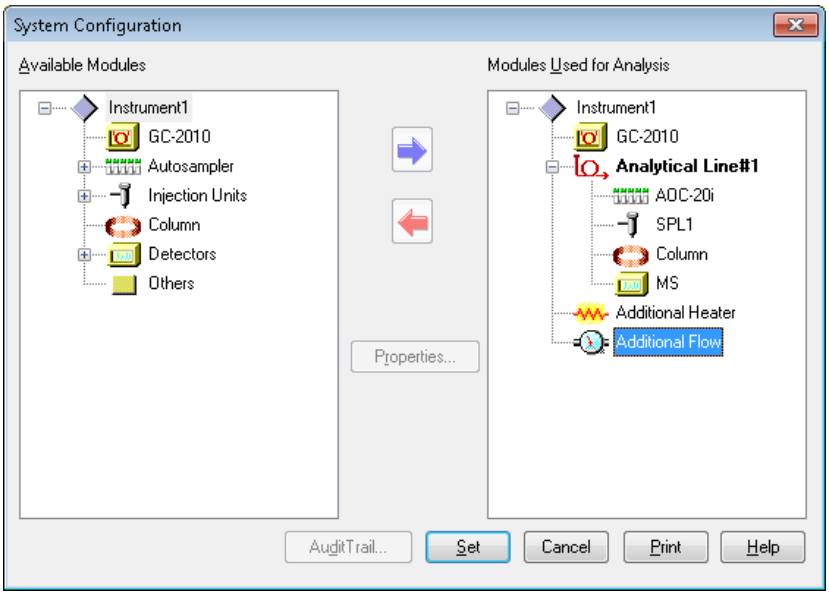
18) Press the [PF3] button to switch flowrate control ON.

19) Press the [COL] button on the GC panel.

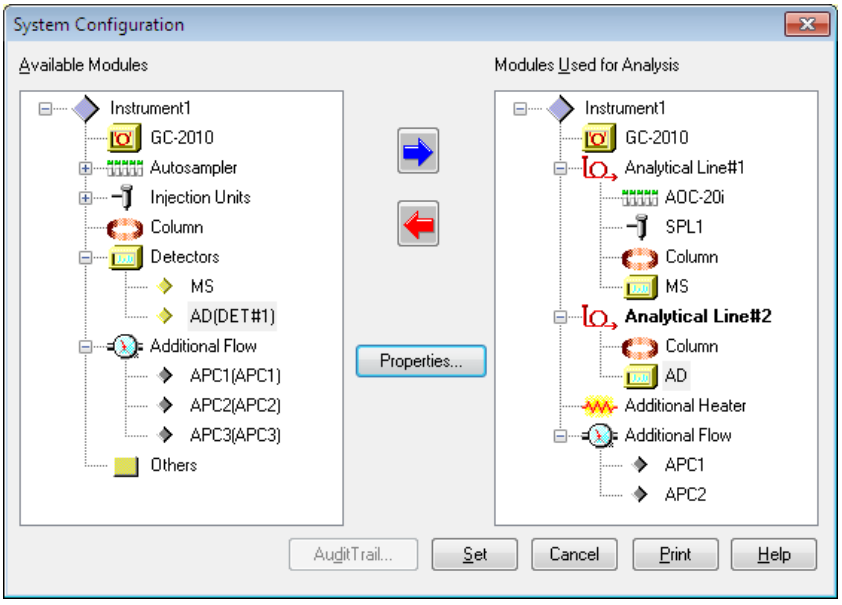
20) Press the [PF3] button to switch [Fan] ON.

21) Specify configuration settings in the [GCMS Real Time Analysis] program. Select [SPL1] and add it to [Modules Used for Analysis]. An example of configuration settings is shown below.

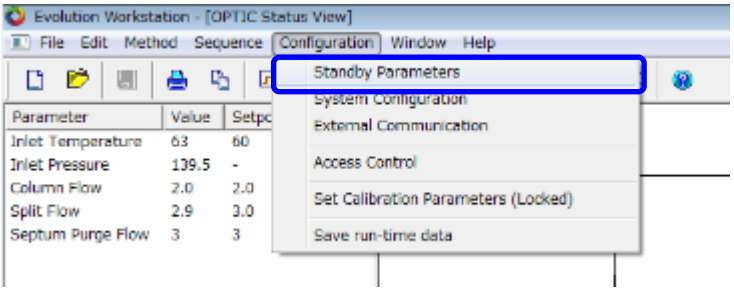
If a Sniffer Is Not Used



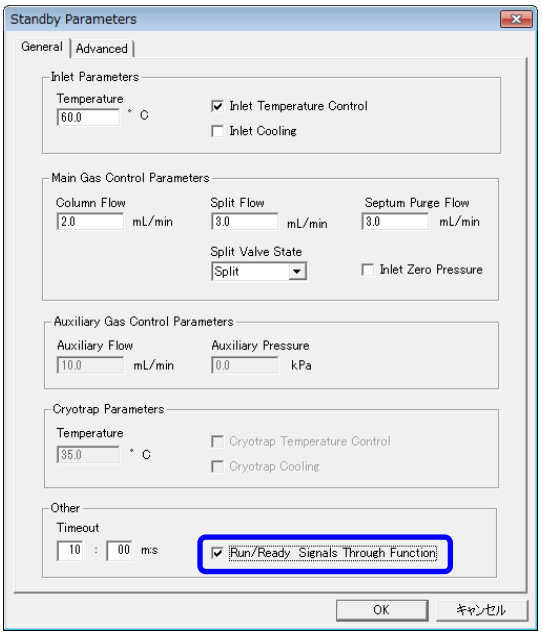
If a Sniffer Is Used



22) In the Evolution Workstation, open the [Standby Parameters] sub-window.

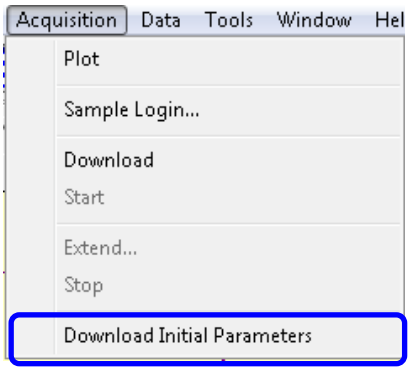


23) In the [Standby Parameters] sub-window, select the [Run/Ready Signals Through Function] checkbox.



24) Click [OK] to close the sub-window.

25) Load the method file and click [Download Initial Parameters] on the [Acquisition] menu.



26) When each parameter value reaches its setting value, the status of both the [GC] and [MS] units is [Ready], then the switch from the OPTIC-4 unit to the SPL unit is finished.

Appendix 8. Analysis Using Monolithic Adsorbent by the OPTIC-4 Unit

Monolithic material sorptive extraction (MMSE) is a pretreatment method that traps and concentrates target components on a disc or rod-shaped monolithic silica adsorbent (MonoTrap), from which the components are removed by solvent extraction or thermal desorption. The trapping material can be either exposed to gases in the sample headspace area or immersed in liquid samples, which makes it easy to collect and concentrate samples. Trapped samples can be measured in the GC/MS unit by injecting a solution of solvent extracted components or by thermal desorption using an OPTIC-4 unit. To use solvent extraction for analysis, follow the procedure in "*2.7 Analyzing Samples*". To use thermal desorption with the OPTIC-4 unit, follow the procedure below.

NOTE

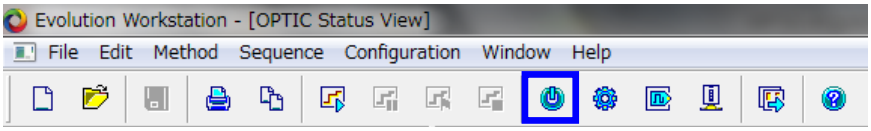
An OPTIC-4 unit is required for analysis by thermal desorption

1) Prepare the MonoTrap material with trapped sample and insert it in a glass tube.

NOTE

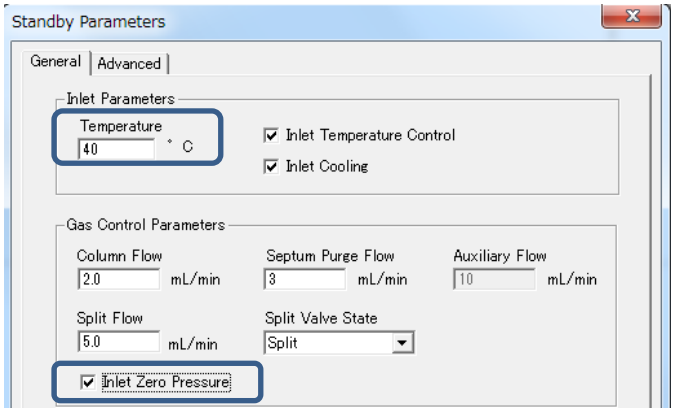
If immersion and shaking was used for MonoTrap sampling, large amounts of the measurement sample may be clinging to the MonoTrap material. Therefore, rinse it off with ultra pure water before inserting it in the glass tube.

2) Confirm that the system status indicated in the [OPTIC Status View] sub-window of the Evolution Workstation is "Standing by."
3) Click [Standby Parameters].

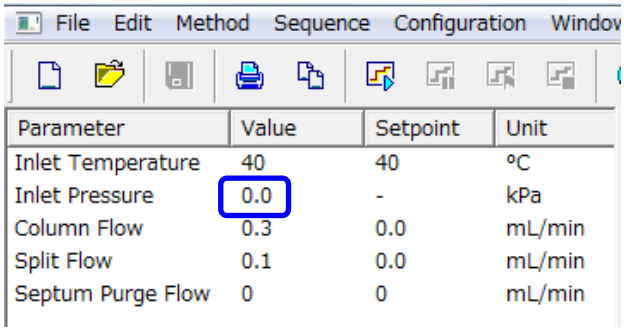


4) Set 40 °C at [Temperature].

5) Select the [Inlet Zero Pressure] checkbox and click [OK].



6) Confirm that the inlet pressure is zero.



7) Loosen the large nut that fastens the head unit to the inlet port and remove the head unit.

8) Lift up on the liner contained inside, which is fitted with an O-ring.

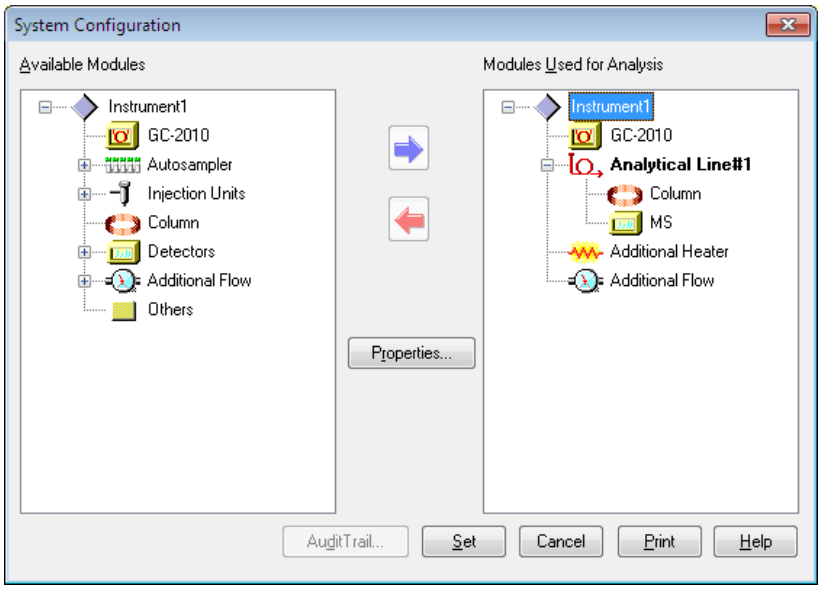
9) Install the O-ring on the glass tube prepared in Step 1) and insert them into the inlet port.

10) Install the head unit and fasten it by hand-tightening the nut.

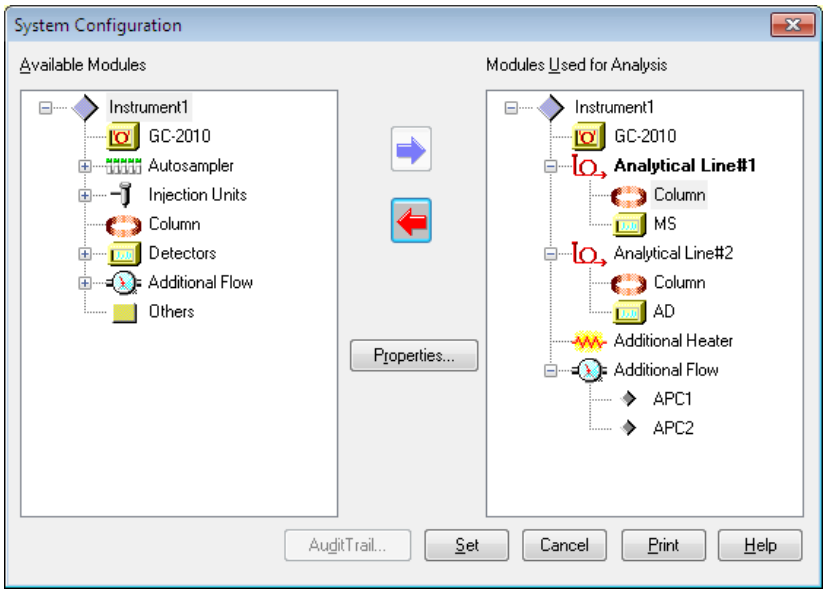
11) Enter the original parameter settings in [Standby Parameters] and click [OK].

12) Specify configuration settings in the [GCMS Real Time Analysis] program. Remove the autosampler from configuration settings. An example of configuration settings is shown below.

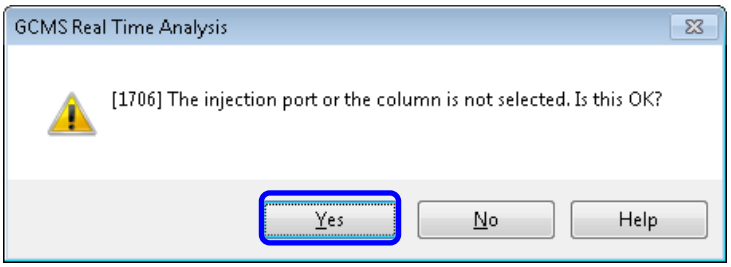
If a Sniffer is Not Used



If a Sniffer is Used



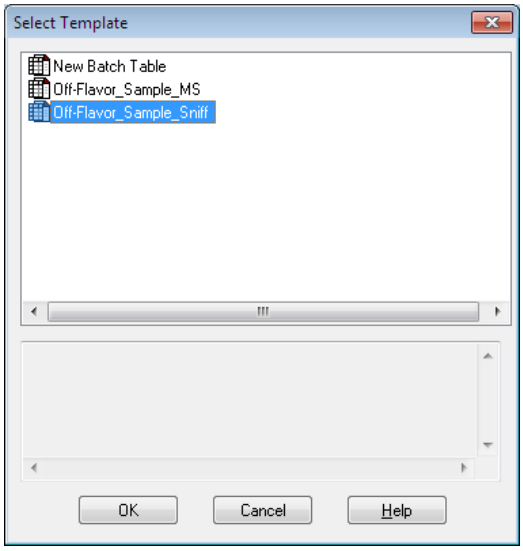
13) When the following sub-window is displayed, click [Yes].



14) Open the method file saved in step 11) of "*2.6 Creating Method Files for Analyzing Samples*" in the [Acquisition] sub-window.

15) Open the [Batch Table] window and click  (New)

16) In the [File New] sub-window, select the template "Off-Flavor_Sample_Sniff" if using a sniffer or "Off-Flavor_Sample_MS" if not using a sniffer.



17) Specify [Sample Name] and [Sample ID] values. Enter "1" in the [Vial#] column. Also specify the method files created in "*2.6 Creating Method Files for Analyzing Samples.*" To include automatically calculated component concentration values in quantitative results, specify [Sample Amt.] and [Dil. Factor] values.

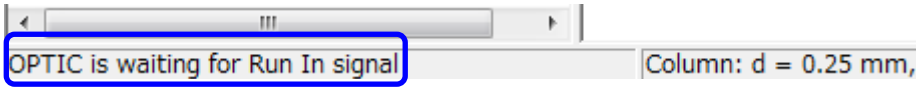
	Vial#	Sample Name	Sample ID	Sample Am	Dil. Fact	Sample Type	Analysis Type	Method File
1-1	1	Sample_A	0001	2	100	0:Unknown	IT QT	5292015_1.qgm
1-2	1	Sample_A	0001	1	1	0:Unknown		5292015_1.qgm

18) Delete rows two onward by highlighting and right-clicking on the rows and clicking [Delete Row] in the right-click menu.

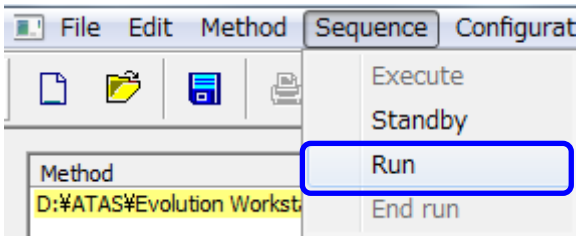
19) Execute realtime batch analysis

20) Execute the OPTIC-4 method according to the procedure described in " steps 7) to 20) in "*2.4.1 Measuring Standard Samples.*"

21) Confirm that the OPTIC-4 unit waits for sample injection.



22) Click [Run] on the [Sequence] menu to start analysis.



Appendix 9. Procedures for registering additional compounds

The procedures for registering additional compounds in the database are as follows.

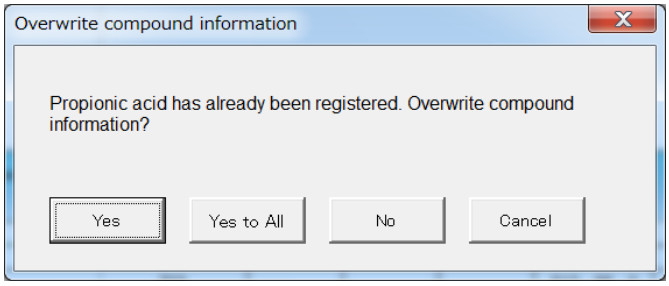
> ⚠ CAUTION
>
> If you make an error in the additional registration procedures, the information registered as default in the database may become corrupted. When registering additional compounds, it is recommended that before starting the registration process, you save a copy of the file under a different name.

> ⚠ CAUTION
>
> With regards to calibration curve information for the compounds additionally registered, analyze a dilution series of a standard sample using the liquid injection method. Then create a calibration curve from the results, and register it in the database.

1) Implement everything up to autotuning, in accordance with the procedures in 2.1 Preparation to 2.3 Specifying Pretreatment Units.
2) Create a quantitative method file for Scan mode, in accordance with the procedures in section 3.3 of the GCMS Operation Guide, Method Development.
   Use a template method file to measure the standard mixture solution of the additionally registered compounds, and use a method file for adjusting retention times to measure the standard n-alkane mixture solution.
3) Register the compound information in the database.
   If you are registering information for Scan mode and SIM mode, refer to the procedures in section 3.4 of the GCMS Operation Guide, Method Development.
   If you are registering information for MRM mode, refer to the procedures in sections 3.5 and 3.6 of the GCMS Operation Guide, Method Development.
   Name and save the database registered, and then close it.

⚠ CAUTION

When registering information in the database, the following message will be displayed if the same compound has already been registered. If you overwrite the registered information, the default information registered in the database may become corrupted. Accordingly, click [No], and skip the compound information displayed in the message, without registering it.



4) Run the postrun analysis program. Open the data file for n-alkanes that was created at step 2).

5) Click 

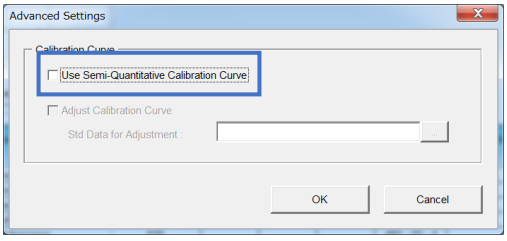
, and open the database saved at step 3).

6) For all the compounds including the additionally registered compounds and the internal standard substances, configure [Target] in the corresponding [Type] column cells.

Serial#	Type	Acq. Mode	ISTD Group	Level1 Conc (IS)	Method No.	Compound Name (E)	Ret. Index 1 (InertCap 5MS/
142	Target	MRM			1	2,4,6-Tribromophenol	1662
143	Target	MRM			1	1-Tetradecanol	1679
144	Target	MRM			1	gamma-Dodecalactone	1686
145	Target	MRM			1	Dibenzyl disulfide	2105
146	Target	MRM			1	p-Bromofluorobenzene	928
147	Target	MRM			1	1,2-Dichlorobenzene-d4	1034
148	Target	MRM			1	Acenaphthene-d10	1500
149	Target	MRM			1	additionally registered compound A	1030
150	Target	MRM			1	additionally registered compound B	1505

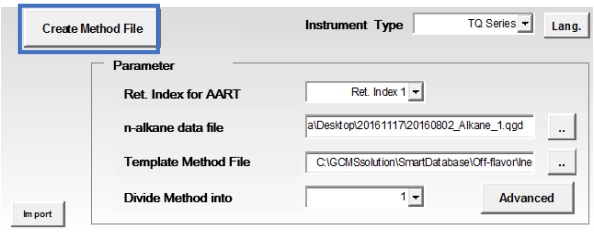
7) Configure [SIM] or [MRM] in the [Acq. Mode] area.

8) Click [Advanced] and clear the [Use Semi-Quantitative Calibration Curve] checkbox.

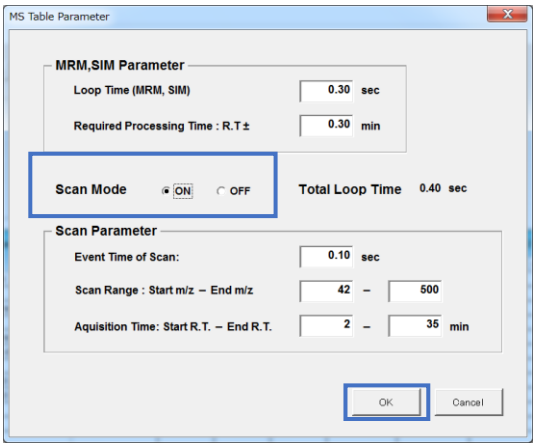


9) Click [OK] to close the sub-window.

10) Click [Create Method File].



11) Select [ON] in the [Scan Mode] area, and click [OK].



12) The [Progress Bar] sub-window is displayed and a method file is automatically created.

13) Name and save the method file.

14) Name and save the database file, and then close the file.

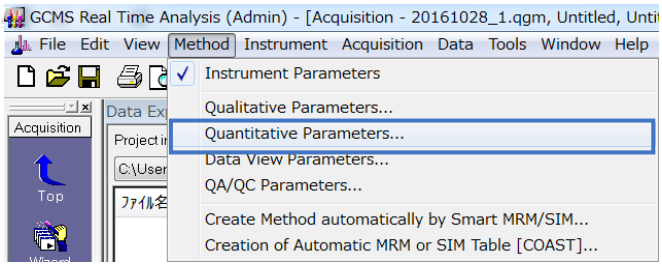
15) Run the analysis program.

16) On the [Real Time] assistant bar, click 

.

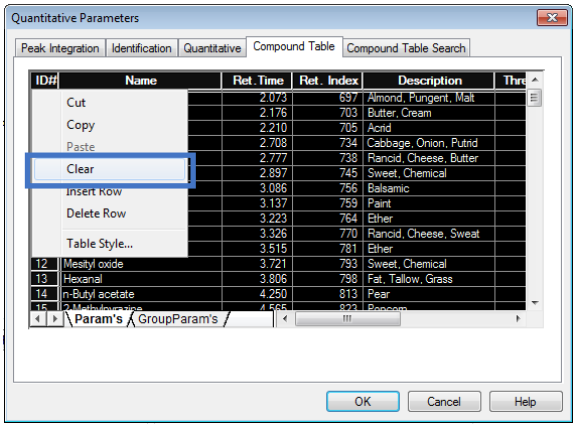
17) Open the method file saved at step 13).

18) Click [Quantitative Parameters] on the [Method] menu.

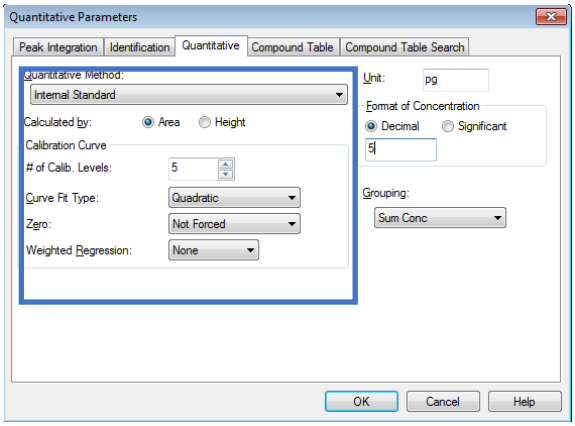


19) Click the [Compound Table] tab.

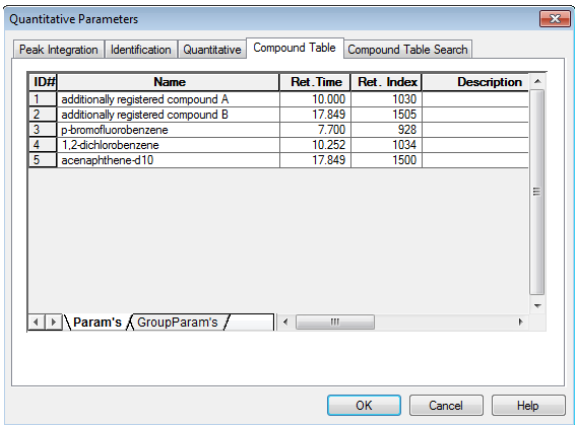
20) Select the rows corresponding to compounds other than the additionally registered compounds and the internal standard substances, and click [Clear].



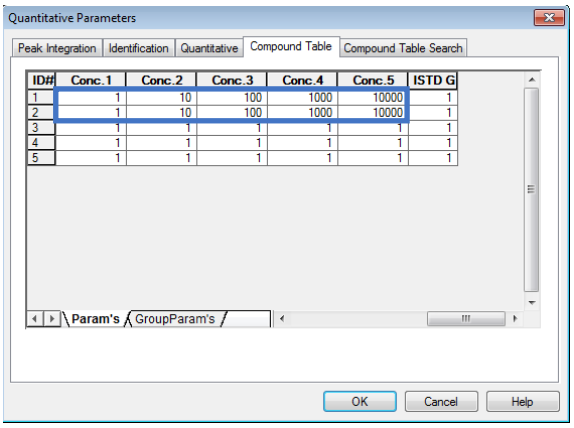
21) On the [Quantitative] tab, configure the parameters in the blue box below.



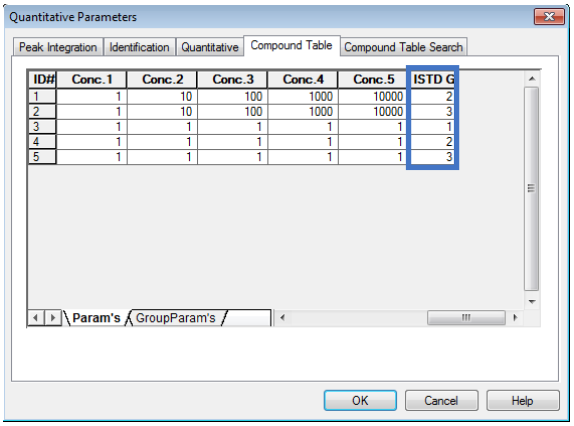
22) Click the [Compound Table] tab.



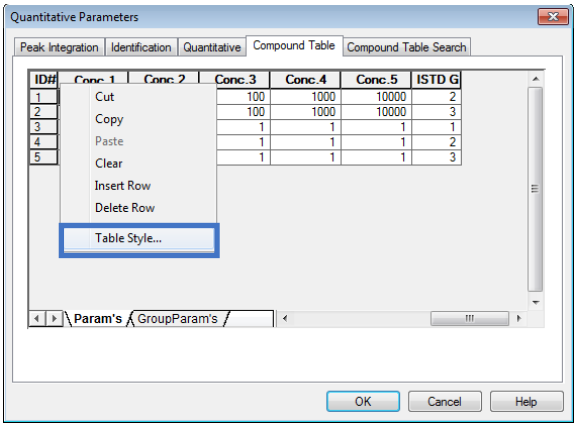
23) In concentration columns (1) to (5) for the additionally registered compounds, configure the values shown below.



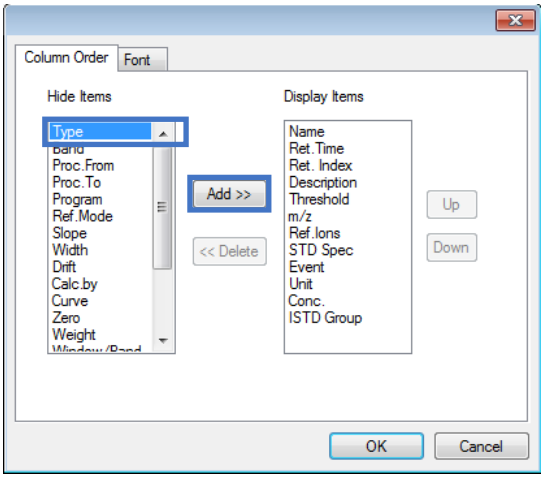
24) Configure the [ISTD Group] column for the additionally registered compounds and the internal standard substances. For the internal standard substances, set p-bromofluorobenzene to "1," 1,2-dichlorobenzene to "2," and acenaphthene-d10 to "3." Match the ISTD group for the additionally registered components to the ISTD group for the internal standard substance with a retention time in the same vicinity.



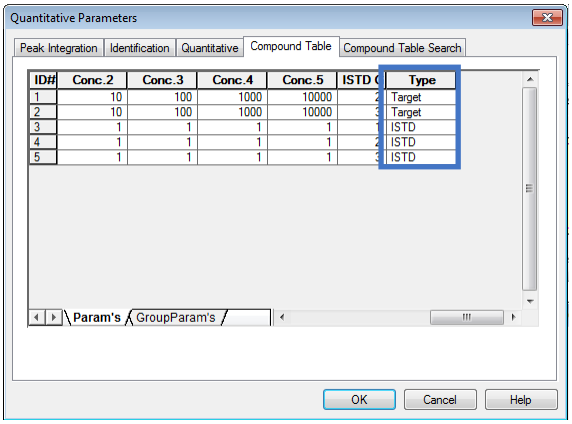
25) Right-click, and select [Table Style].



26) Add [Type] to the items displayed. Then click [OK] and close the sub-window.



27) Configure the cells in the [Type] column corresponding to the internal standard substances to [ISTD].



28) Click [OK] to close the sub-window.

29) Save the method file.

30) Prepare a dilution series for the standard sample for the additionally registered compounds. Ensure that the internal standard substances have been prepared with the concentrations noted below.

4-Bromofluorobenzene	: 0.1 µg/mL
1,2-Dichlorobenzene-d4	: 0.1 µg/mL
Acenaphthene-d10	: 0.1 µg/mL

Note that the following are recommended as the standard samples for the internal standard substances.

Internal Standard/Surrogate Standard Mixture Mix EPA524.1、EPA524.2 Cat: 47358-U
(2000 µg/mL 4-Bromofluorobenzene, 1,2-Dichlorobenzene-d4, Fluorobenzene in methanol) Supplier: Sigma-Aldrich
*1: Fluorobenzene is included in the standard sample, but is not a target component.

Acenaphthene-d10 solution Cat: 48417
(2000 µg/mL component in methylene dichloride) Supplier: Sigma-Aldrich

31) Place the standard samples prepared at step 30) into the autosampler.

32) On the [Real Time] assistant bar, click the 

icon to display the [Batch Table] window.

33) Click  (New) on the toolbar.

In the [File New] sub-window, select "Off-Flavor_Sample_Sniff" if using a sniffer or "Off-Flavor_Sample_MS" if not using a sniffer.

34) Create the batch table. Specify the method file created at step 29) in the [Method File] column, and set the injection quantity to 1 µL.

	Vial#	Sample Name	Sample ID	Sample Amt.	Dil. Factor	Sample Type	Analysis Type	Method File	Data File	Level#	Inj. Volume
1	1	STD	1ppb	1	1	1:Standard(I)	IT QT	test.qgm		1	1
2	2	STD	10ppb	1	1	1:Standard	IT QT	test.qgm		2	1
3	3	STD	100ppb	1	1	1:Standard	IT QT	test.qgm		3	1
4	4	STD	1ppm	1	1	1:Standard	IT QT	test.qgm		4	1
5	5	STD	10ppm	1	1	1:Standard	IT QT	test.qgm		5	1

35) If you are using the AOC-5000 Plus, enter the tray name in the [Tray] column. If you are using the AOC-6000, enter the rack name in the [Tray] column.

36) Delete any rows unnecessary for measurements by highlighting and right-clicking on the rows and clicking [Delete Row] on the right-click menu.

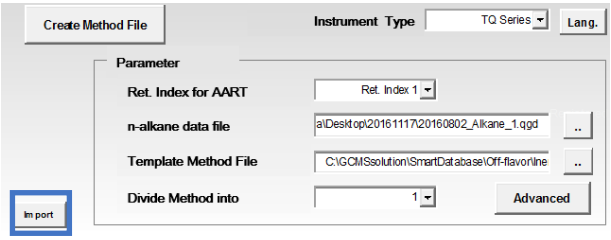
37) Name and save the batch file.

38) Execute the realtime batch analysis.

39) In accordance with the procedures in section 5.3.1 of the GCMS Operation Guide, Basic Operation Guide, confirm and revise the calibration curve, and then save the method file.

40) Open the database file saved at step 14).

41) Click [Import].

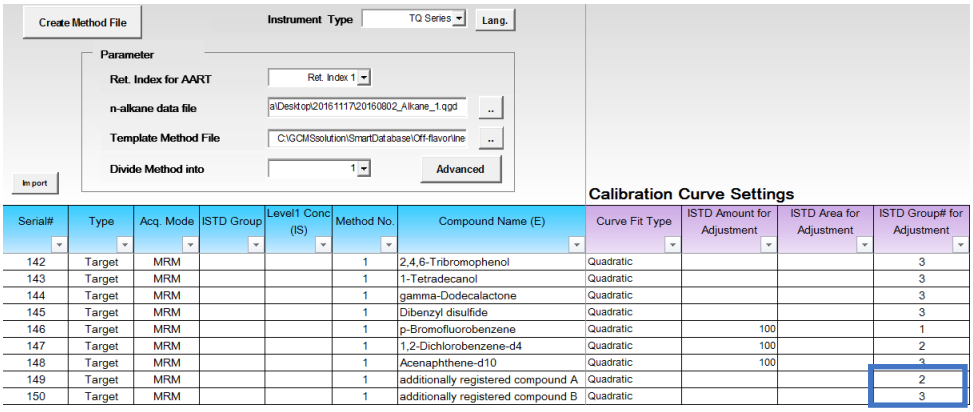


42) Select the method file saved at step 39).

43) A message related to overwriting compound information will be displayed. Click [No] for the internal standard substances, and click [Yes] for the additionally registered compounds.

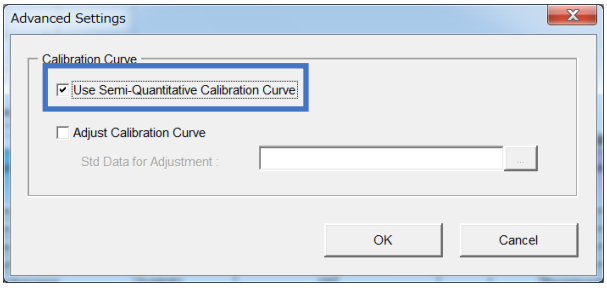
44) The calibration curve information will now be imported.

45) In the [ISTD Group# for Adjustment] column for the additionally registered compounds, configure the ISTD group set at step 24).



Serial#	Type	Acq. Mode	ISTD Group	Level1 Conc (IS)	Method No.	Compound Name (E)	Curve Fit Type	ISTD Amount for Adjustment	ISTD Area for Adjustment	ISTD Group# for Adjustment
142	Target	MRM			1	2,4,6-Tribromophenol	Quadratic			3
143	Target	MRM			1	1-Tetradecanol	Quadratic			3
144	Target	MRM			1	gamma-Dodecalactone	Quadratic			3
145	Target	MRM			1	Dibenzyl disulfide	Quadratic			3
146	Target	MRM			1	p-Bromofluorobenzene	Quadratic	100		1
147	Target	MRM			1	1,2-Dichlorobenzene-d4	Quadratic	100		2
148	Target	MRM			1	Acenaphthene-d10	Quadratic	100		3
149	Target	MRM			1	additionally registered compound A	Quadratic			2
150	Target	MRM			1	additionally registered compound B	Quadratic			3

46) Click [Advanced] and select the [Use Semi-Quantitative Calibration Curve] checkbox.



47) Click [OK] to close the sub-window.

48) Ensure that the cells in the [Type] column corresponding to the internal standard substances are blank.

Type	Acq. Mode	ISTD Group	Level1 Conc (IS)	Method No.	Compound Name (E)
Target	MRM			1	2,4,6-Tribromophenol
Target	MRM			1	1-Tetradecanol
Target	MRM			1	gamma-Dodecalactone
Target	MRM			1	Dibenzyl disulfide
	MRM			1	p-Bromofluorobenzene
	MRM			1	1,2-Dichlorobenzene-d4
	MRM			1	Acenaphthene-d10
Target	MRM			1	additionally registered compound A
Target	MRM			1	additionally registered compound B

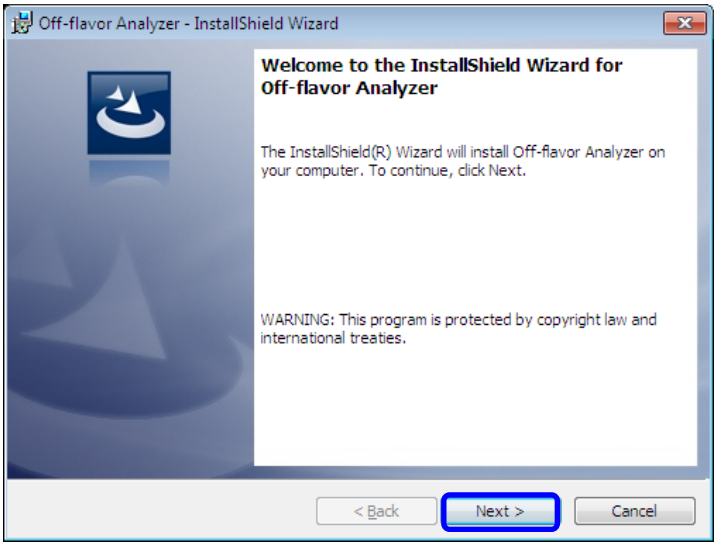
49) Overwrite the Smart Database file and close the file.

Appendix 10. Software Installation

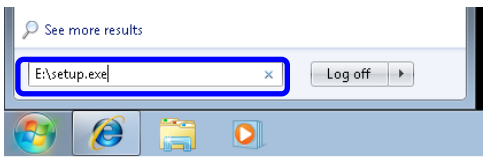
Install Software in accordance with the following procedures. Explanation in this section is given for a Windows 7 based computer.

> **NOTE**
>
> To install the database, GCMSsolution Ver.4.31 needs to be installed in advance.

1) Insert the database installation disk into the DVD-ROM drive. The installer starts up automatically. Click [Next].

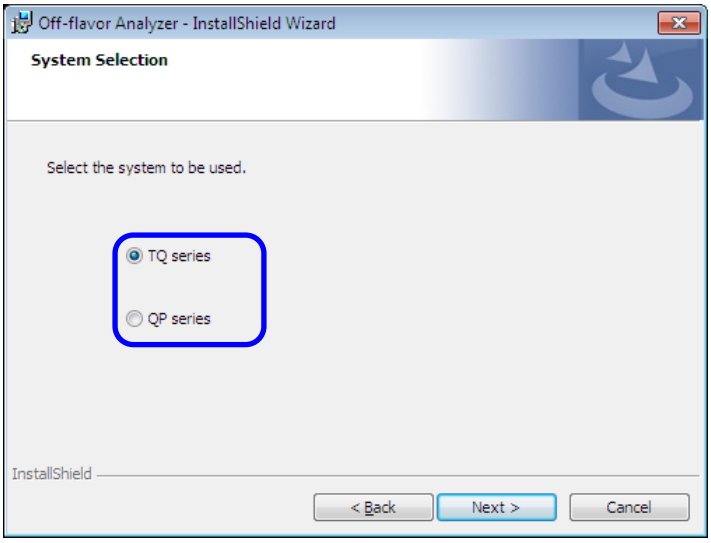


> **NOTE**
>
> If it does not, click the Windows Start menu. Enter "E:\setup.exe," and press the [Enter] key. (Where E: is DVD-ROM drive)

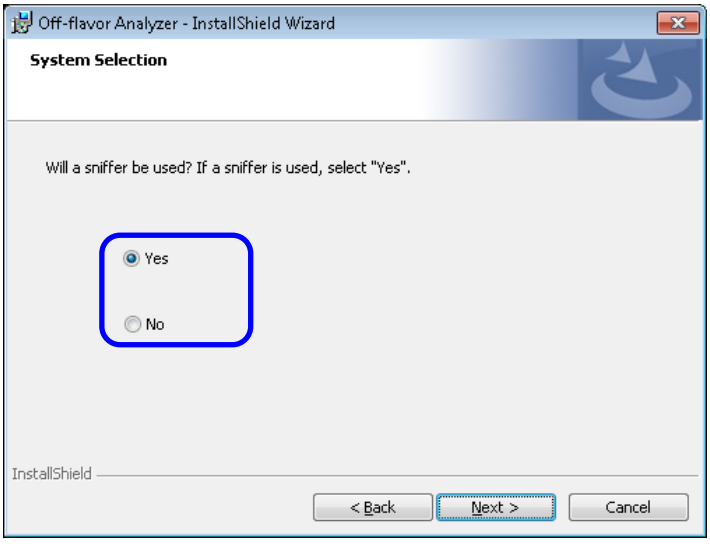


> **NOTE**
>
> The dialog box "Do you want to allow the following program to make changes to this computer?" may be displayed. In this case, click the [Yes] to proceed.

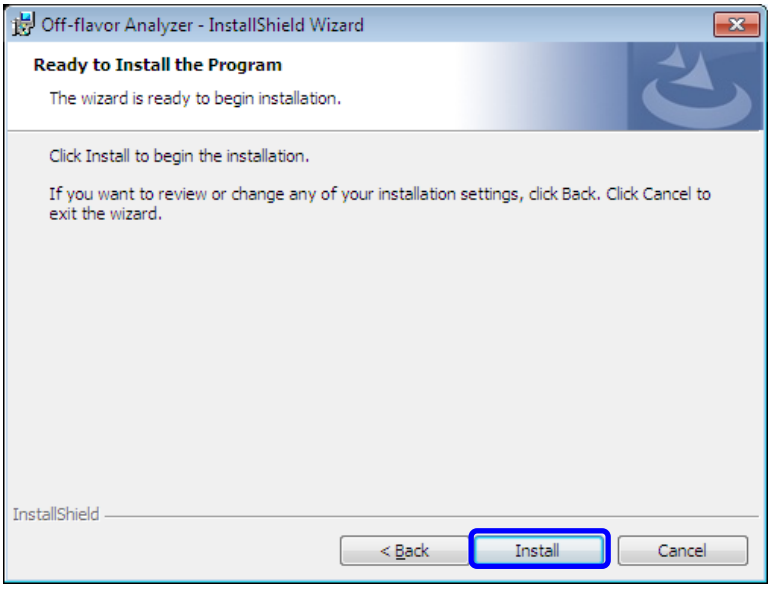
2) Select the type of GC/MS system used.
Select [QP series] if GCMS-QP2010 Ultra or GCMS-QP2020systems are used.
Select [TQ series] if GCMS-TQ8030 or GCMS-TQ8040 systems are used.
Click [Next].



3) Select whether or not a sniffer is used. Click [Next].



4) Click [Install] in the [Ready to Install the Program] sub-window. Every files are installed in the designated locations.



Nomally, every files are installed in the
“C:¥GCMSsolution¥Data¥Smart Database¥Off-flavor” folder

5) When installation completes, the [InstallShield Wizard Completed] sub-window opens. Click [Finish] to complete the installation.