225-45218B
Nov. 2024

GC-MS QP and TQ Series

Smart Database

Smart Aroma Database 1.8

Instruction Manual 1.8

For GCMSsolution

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

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Introduction

Read this Instruction Manual thoroughly before using the product.

Thank you for purchasing this product. This manual describes 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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Indications Used in This Manual

Throughout the text in this manual, warnings and other information essential when using this unit, such as cautionary or prohibited items, appear classified as per the following:

Indication	Meaning
⚠ 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	Indicates information provided to improve product performance.
Reference	Indicates the location of related reference information.

⚠ WARNING

When the customer uses the CD-ROM

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

Product Warranty

Shimadzu provides the following warranty for this product.

1. Warranty Period — Please contact your Shimadzu representative for information about the period of this warranty.
2. Description: — If a product/part failure occurs for reasons attributable to Shimadzu during the warranty period, Shimadzu will repair or replace the product/part free of charge. However, in the case of products which are usually available on the market only for a short time, such as personal computers and their peripherals/parts, Shimadzu may not be able to provide identical replacement products.
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   2) In no event will Shimadzu's liability to you, whether in contract, tort (including negligence), or otherwise, exceed the amount you paid for the product.
4. Exceptions — Failures caused by the following are excluded from the warranty, even if they occur during the warranty period.
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   2) Repairs or modifications performed by parties other than Shimadzu or Shimadzu designated companies
   3) Product use in combination with hardware or software other than that designated by Shimadzu
   4) Computer viruses leading to device failures and damage to data and software, including the product's basic software
   5) Power failures, including power outages and sudden voltage drops, leading to device failures and damage to data and software, including the product's basic software
   6) Turning OFF the product without following the proper shutdown procedure leading to device failures and damage to data and software, including the product's basic software
   7) Reasons unrelated to the product itself
   8) Product use in harsh environments, such as those subject to high temperatures or humidity levels, corrosive gases, or strong vibrations
   9) Fires, earthquakes, or any other act of nature, contamination by radioactive or hazardous substances, or any other force majeure event, including wars, riots, and crimes
   10) Product movement or transportation after installation
   11) Consumables and equivalent items
   Recording media such as are considered consumable items.

- 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.

Contents

1 Smart Aroma Database

1.1 Overview

Optimizing columns and analytical conditions and gathering GC/MS information for compounds involved in analyzing the odors/aromas of foods, beverages, chemical products, or other samples is an extremely tedious and time-consuming process. Using non-targeted analysis to comprehensively analyze samples, without specifying specific compounds to analyze, can cause problems with tedious data analysis and inaccurate compound identification results.

1.8 The Smart Aroma Database offers method files and database files with a wealth of information registered about columns, analytical conditions, and compounds optimized for a wide variety of odor/aroma components.

Method files for scan mode analysis include retention index values determined from n-alkanes, so that the

1.8 automatic adjustment of retention time (AART) function can be used with a standard sample to easily adjust retention times for registered compounds. That means target compounds can be efficiently identified based on the registered retention time, mass chromatogram, and mass spectrum information.

As a “Smart Database,” retention times adjusted using the AART retention time adjustment function can be used to create methods for high-sensitivity targeted SIM or MRM analysis.

Furthermore, database files are saved in Excel format to enable editing or adding compounds.

> **NOTE**
>
> This instruction manual has been written assuming the reader already has a basic knowledge about using GC/MS, GC-MS/MS, and GCMSsolution products. In many instances, explanations include GCMSsolution-specific nomenclature and terminology. If using a GC/MS or GC-MS/MS system or the GCMSsolution software for the first time or if such terminology is not clear, then also refer to the corresponding GC/MS or GC-MS/MS instruction manual, the GCMS Operation Guide, and the help menu in GCMSsolution.

1.2 Operating Environment

Use the database in the following operating environment.

Operating System	Microsoft Windows 11 Professional 64-bit version Microsoft Windows 10 Professional 64-bit version Microsoft Windows 7 Professional SP1 32 or 64-bit version
Excel	Microsoft Excel2021 (32 or 64-bit version) Microsoft Excel2019 (32 or 64-bit version) Microsoft Excel2016 (32-bit version)
Workstation Software	GCMSsolution Ver. 4.53 SP1 or later

1.3 Supported Models

This product can be used with the following systems.

- GCMS-QP series: GCMS-QP2020 series or GCMS-QP2010 SE
- GCMS-TQ series: GCMS-TQ series

This product can be used with the following columns.

- SH-I-5Sil MS (30m x 0.25mm I.D. df=0.25 μm) P/N: 221-75954-30
  Contact: Shimadzu Corporation
  This slightly polar column is suitable for targeting mainly compounds with relatively low polarity, such as terpenes.

- SH-PolarWax (60m x 0.25mm I.D. df=0.25 μm) P/N: 227-36247-02
  Contact: Shimadzu Corporation
  This polar column is suitable for targeting mainly compounds with high polarity, such as organic acids. Due to the GC conditions used that prioritize separation, this column is ideal for comprehensive analysis.

- InertCap PureWAX (30m x 0.25mm I.D. df=0.25 μm) P/N: 1010-68142
  Contact: GL Sciences Inc.
  This polar column is suitable for targeting mainly compounds with high polarity. It is the same column as used in GC/MS Off-Flavor Analyzer, so it can be used for both systems.

NOTE

In order to use the retention index values registered in the database, use a column with the specified liquid phase type, length, internal diameter, and film thickness that is made by the specified manufacturer.

NOTE

Due to differences between column production lots of Wax column, retention index values can vary. If that occurs, use a standard sample to adjust the retention times based on the retention times predicted from the registered retention index values. (Refer to Appendix 10 Retention Time Adjustment Using a Standard Sample.)

In addition to AOC-20 and AOC-30 series autosamplers typically used for liquid injection, the database also supports the following pretreatment systems. For more information about the procedures for respective pretreatment systems, refer to Appendices 4 to 7.

- AOC-6000 series
- HS-20 series
- TD-30 series
- OPTIC-4

1.4 Product Contents

1.4.1 Product Contents

This product includes a CD-ROM. If it is missing, contact a Shimadzu representative.

P/N	Name	Quantity
225-50791-91	CD-ROM	1

The CD-ROM includes an installer program and a PDF version of the instruction manual.

NOTE

Though the product does not include a column, be sure to use the specified type.

1.4.2 Method File Details

Executing the installer program (setup.exe) included with the product installs the following method files.

During installation, select the type of instrument (QP or TQ) so that files for the given instrument type are installed. (In this case, “C:” refers to the drive where GCMSsolution is installed.)

Files for the applicable columns are installed in the following folders for respective columns.

For SH-I-5Sil MS:

“C:\GCMSsolution\SmartDatabase\Aroma\SH-I -5Sil_MS” folder

For SH-PolarWax:

“C:\GCMSsolution\SmartDatabase\Aroma\SH-PolarWax” folder

For InertCap PureWax:

“C:\GCMSsolution\SmartDatabase\Aroma\InertCap_Pure-Wax” folder

QP Methods

Column	File Name	Description
SH-I-5Sil MS	Aroma_QP_SH-I-5SilMS_Scan.qgm	Method file for analyzing aromas/odors (Scan)
	Aroma_QP_SH-I-5SilMS_AART.qgm	Method file for retention time adjustment
	Aroma_QP_SH-I-5SilMS_Correct.qgm	Method file for sensitivity correction analysis
	Aroma_QP_SH-I-5SilMS_Template.qgm	Template for creating SIM method file from Smart Database
SH-PolarWax	Aroma_QP_SH-PolarWax _Scan.qgm	Method file for analyzing aromas/odors (Scan)
	Aroma_QP_SH-PolarWax_AART.qgm	Method file for retention time adjustment
	Aroma_QP_SH-PolarWax_Correct.qgm	Method file for sensitivity correction analysis
	Aroma_QP_SH-PolarWax_Template.qgm	Template for creating SIM method file from Smart Database
InertCap PureWAX	Aroma_QP_IC-WAX_Scan.qgm	Method file for analyzing aromas/odors (Scan)
	Aroma_QP_IC-WAX_AART.qgm	Method file for retention time adjustment
	Aroma_QP_IC-WAX_Correct.qgm	Method file for sensitivity correction analysis
	Aroma_QP_IC-WAX_Template.qgm	Template for creating SIM method file from Smart Database

TQ Methods

Column	File Name	Description
SH-I-5Sil MS	Aroma_TQ_SH-I-5SilMS_Scan.qgm	Method file for analyzing aromas/odors (Scan)
	Aroma_TQ_SH-I-5SilMS_AART.qgm	Method file for retention time adjustment
	Aroma_TQ_SH-I-5SilMS_Correct.qgm	Method file for sensitivity correction analysis
	Aroma_TQ_SH-I-5SilMS_Template.qgm	Template for creating SIM or MRM method file from Smart Database
SH-PolarWax	Aroma_TQ_SH-PolarWax_Scan.qgm	Method file for analyzing aromas/odors (Scan)
	Aroma_TQ_SH-PolarWax_AART.qgm	Method file for retention time adjustment
	Aroma_TQ_SH-PolarWax_Correct.qgm	Method file for sensitivity correction analysis
	Aroma_TQ_SH-PolarWax_Template.qgm	Template for creating SIM or MRM method file from Smart Database
InertCap PureWAX	Aroma_TQ_IC-WAX_Scan.qgm	Method file for analyzing aromas/odors (Scan)
	Aroma_TQ_IC-WAX_AART.qgm	Method file for retention time adjustment
	Aroma_TQ_IC-WAX_Correct.qgm	Method file for sensitivity correction analysis
	Aroma_TQ_IC-WAX_Template.qgm	Template for creating SIM or MRM method file from Smart Database

Hint

For systems with sample flow split for detection by a sniffer unit and MS, use the method files in the folders indicated below. For more details, refer to Appendix 8 Connecting to an Odor Sniffer.

“C:\GCMSsolution\SmartDatabase\Aroma\<column name>\Sniff” folder

1.4.3 Contents of Smart Database

Executing the installer program (setup.exe) included with the product installs the following Smart Database files.

During installation, select the type of instrument (QP or TQ) so that files for the given instrument type are installed.

File location: “C:\GCMSsolution\SmartDatabase”

QP Methods

Column	File Name	Description
SH-I-5Sil MS	SmartDatabase_Aroma_QP_SH-I-5SilMS xlsm	Smart Database file for SH-I-5Sil MS
SH-PolarWax	SmartDatabase_Aroma_QP_SH-PolarWax.xlsm	Smart Database file for SH-PolarWax
InertCap PureWAX	SmartDatabase_Aroma_QP_IC-WAX.xlsm	Smart Database file for InertCap PureWAX

TQ Methods

Column	File Name	Description
SH-I-5Sil MS	SmartDatabase_Aroma_TQ_SH-I-5SilMS xlsm	Smart Database file for SH-I-5Sil MS
SH-PolarWax	SmartDatabase_Aroma_TQ_SH-PolarWax.xlsm	Smart Database file for SH-PolarWax
InertCap PureWAX	SmartDatabase_Aroma_TQ_IC-WAX.xlsm	Smart Database file for InertCap PureWAX

1.4.4 Library Contents

Executing the installer program (setup.exe) included with the product installs the following library files.

File location: “C:\GCMSsolution\library”

Column	File Name	Description
SH-I-5Sil MS	AROMA_5MS	Library file for SH-I-5Sil MS
SH-PolarWax	AROMA_POLARWAX	Library file for SH-PolarWax
InertCap PureWAX	AROMA_ICWAX	Library file for InertCap PureWAX

Each library comprises the following seven files. The following example is for the “AROMA.LIB_5MS” library.

File Name	Description
AROMA_5MS.LIB	Mass spectral library file
AROMA_5MS.FOM	Includes composition formula information for registered components.
AROMA_5MS.SPC	Includes spectral information for registered components.
AROMA_5MS.NAM	Includes name information for registered components.
AROMA_5MS.COM	Includes comment information.
AROMA_5MS.FLG	Includes information about whether or not to target the component in searches.
AROMA_5MS.ADD	Includes links to structural information for registered components

- In Shimadzu GC/MS software, all libraries are indicated in terms of the “*.LIB” file. Operating the software does not require an awareness of the other six files. However, note that the library will not function properly unless all seven files are present in the same library folder.

- The file structure for these libraries is configured as a private library. The file structure is different than for public libraries.

- These libraries include information about structural formulas. Structural formula files are included in the “Aroma_Structure” folder. The structural formula will not function properly unless these folders are present in the following folder.
  File location: “C:\GCMSsolution\library”

1.4.5 Instruction Manual Included

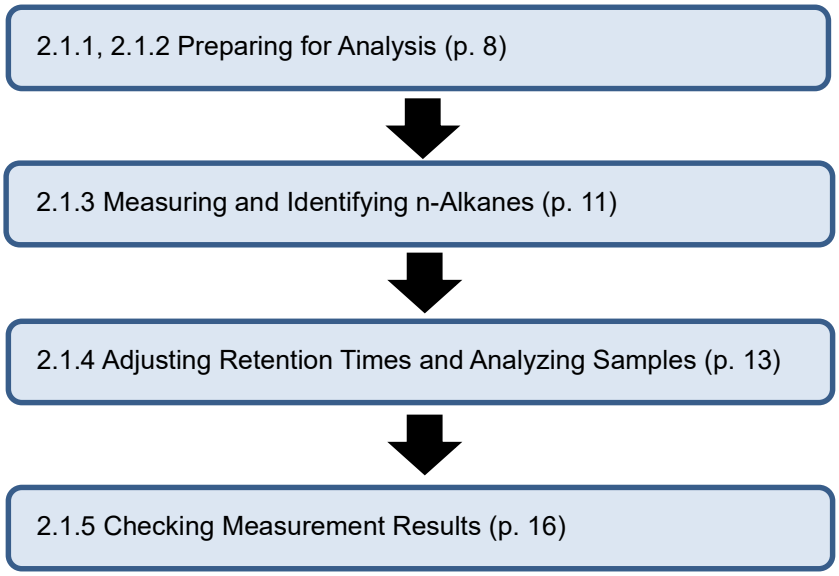
Executing the installer program (setup.exe) included with the product installs the following PDF file of the instruction manual.
The instruction manual file location: C:\GCMSsolution\Manual\SmartDatabase\Aroma

File Name	Description
Smart Aroma Database_Manual.pdf	Smart Aroma Database Instruction Manual

2 Operating Procedure

2.1 Analysis Process Flow Using a Scan Method File

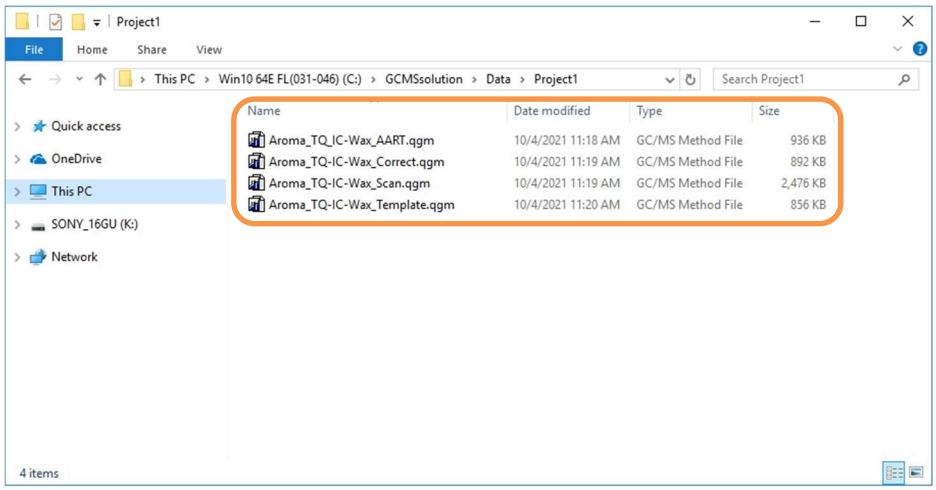
Follow the steps below to analyze samples using a scan method file included in the database. The procedure is basically the same for both QP and TQ type systems, but some of the steps could vary depending on the pretreatment system used. For more information about analysis steps using each type of pretreatment system, refer to the corresponding chapter in appendices.



2.1.1 Creating a Folder for Analysis

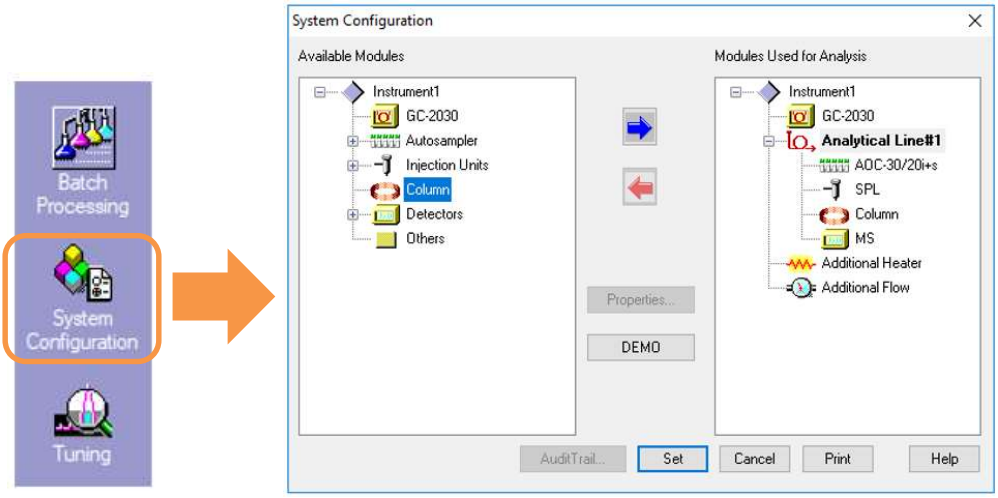
1) Create a folder for saving acquired data files.
(Example: “C:\GCMSsolution\Data\Project1”)

2) Copy the method file that is stored in the “C:\GCMSsolution\SmartDatabase\ Aroma\<column name>” folder and paste it into the folder that was created in step 1.



2.1.2 Setting Instrument Parameters

1) Specify system configuration settings as follows.
   In the [Modules Used for Analysis] field, specify the pretreatment unit to use for analysis line 1.

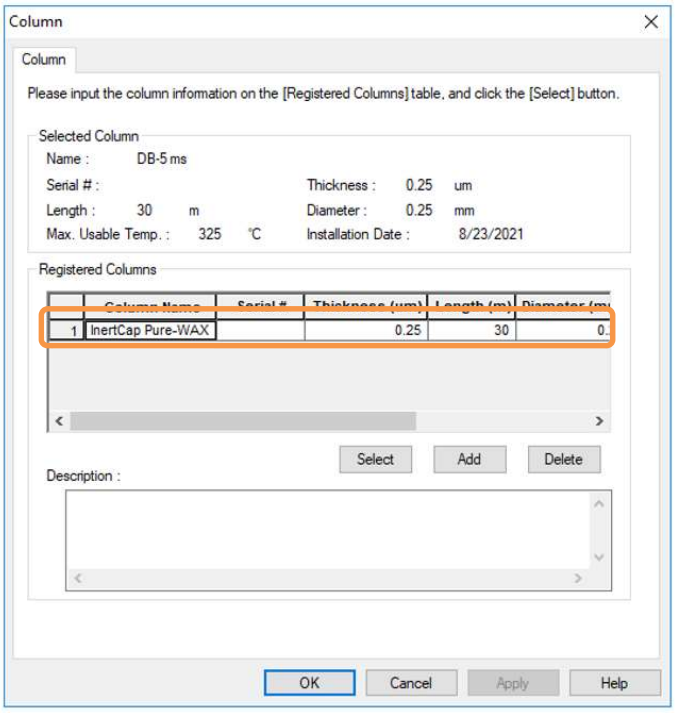


NOTE

Instrument configuration settings differ depending on the pretreatment unit. Refer to the instruction manual for the pretreatment unit to be used.

2) Select the column based on the method file used for analysis and install it in the instrument. Refer to 1.4.2 Method File Details regarding the relationship between methods and columns.

3) In the [System Configuration] window, double-click the [Column] icon for the unit to be used for analysis. Enter information for the column to be used in column property settings.



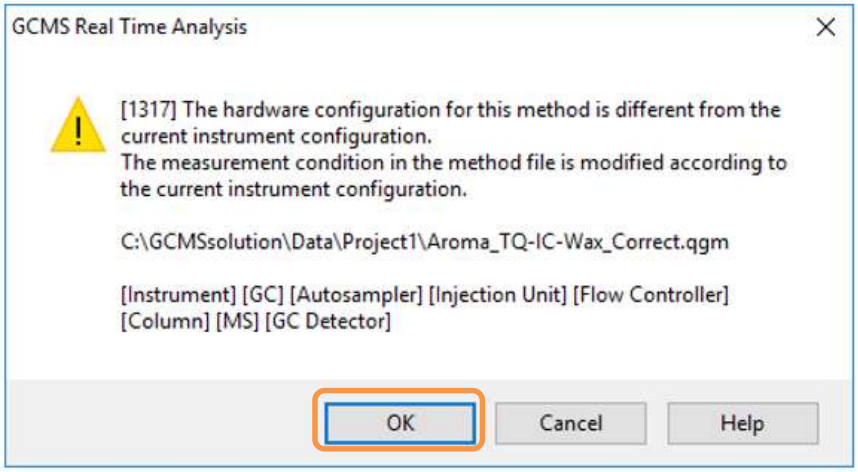
4) Start up the vacuum system and perform auto-tuning.

NOTE

Tune the system whenever the system has been temporarily shut OFF, such as after replacing a column. Even if the system was not shut OFF, it is recommended that the system be retuned about once a month.

NOTE

If the confirmation 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 a method file is loaded in the [GCMS Real Time Analysis]-[Acquisition] window, click [OK].



2.1.3 Measuring and Identifying n-Alkanes

Measure an n-alkane standard mixture solution so that retention times can be adjusted for each compound registered in the method file. Adjust the registered retention times for measurement target compounds based on the n-alkane identification results and retention index values for measurement target compounds (AART function).

Hint

The following n-alkane standard mixture sample is recommended.

C7 - C30 Saturated Alkanes :

SIGMA-ALDRICH Cat#: 49451-U

(1000 µg/mL each component in hexane)

CAUTION

Dilute standard samples under a fume hood or other exhaust system.

<Preparing n-Alkane Standard Mixture Samples>
Prepare the standard mixture sample by diluting C7 - C30 Saturated Alkanes solution (1000 µg/mL) with hexane to a concentration of 50 µg/mL.

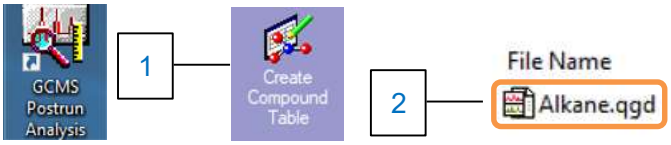
Preparation Example:
Use a microsyringe to place 500 µL of C7 - C30 Saturated Alkanes solution (1000 µg/mL) in a 10 mL volumetric flask and then fill the flask to volume with hexane. Keep the prepared solution stored in a refrigerator or freezer until ready for use.

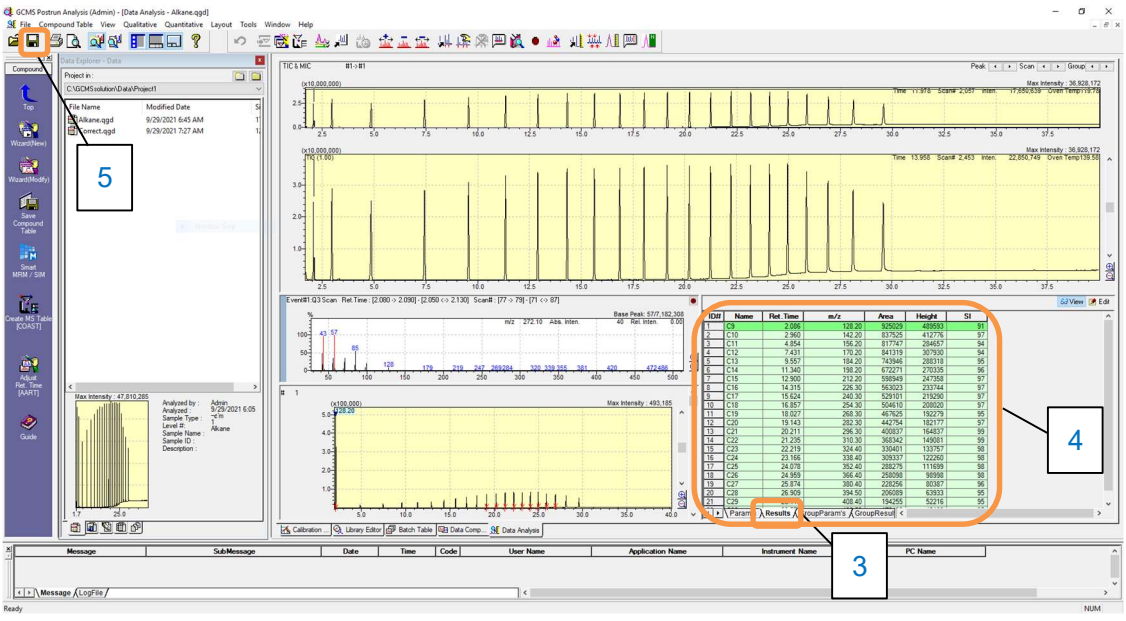
1) Analyze the n-alkane solution (50 µg/mL) using the method file for adjusting retention times.

NOTE

When AOC-30/20 series, AOC-6000 series or OPTIC-4 is used, measure n-alkane is measured by liquid injection (1 µL injection). For the TD-30 series, add n-alkane to the TD tube, and for the HS-20 series, add n-alkane to the HS vial for analysis. Refer to "Appendix 3" to "Appendix 7" for the settings of each auto sampler.

2) Identify the n-alkanes.



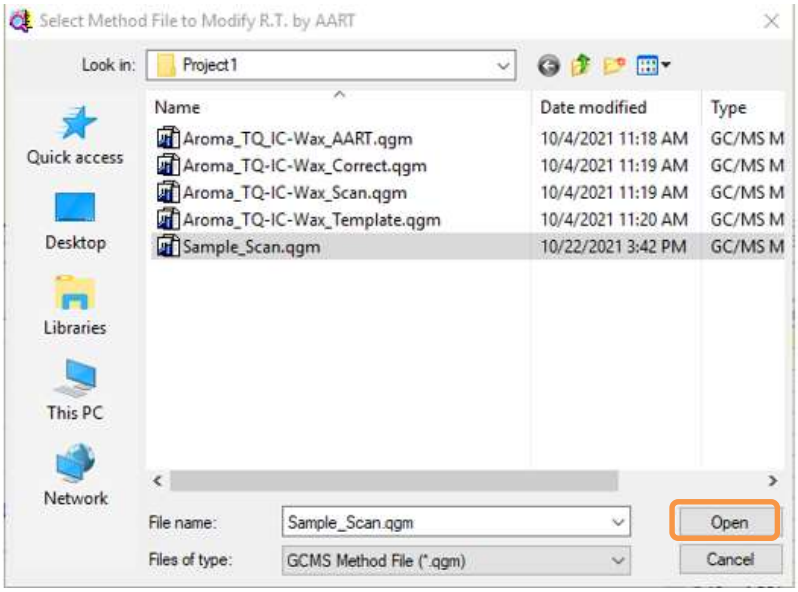


1. Start the [GCMS Postrun Analysis] program and then click the [Create Compound Table] icon on the [Postrun] assistant bar.
2. Open Data Explorer and double-click the icon for the n-alkane measurement data.
3. Click the [Results] tab below the compound table on the left.
4. Identify each component by checking the mass chromatogram (MC) in the quantitation view. If the components are not identified correctly, use manual peak integration.
5. Save the data file by overwriting.

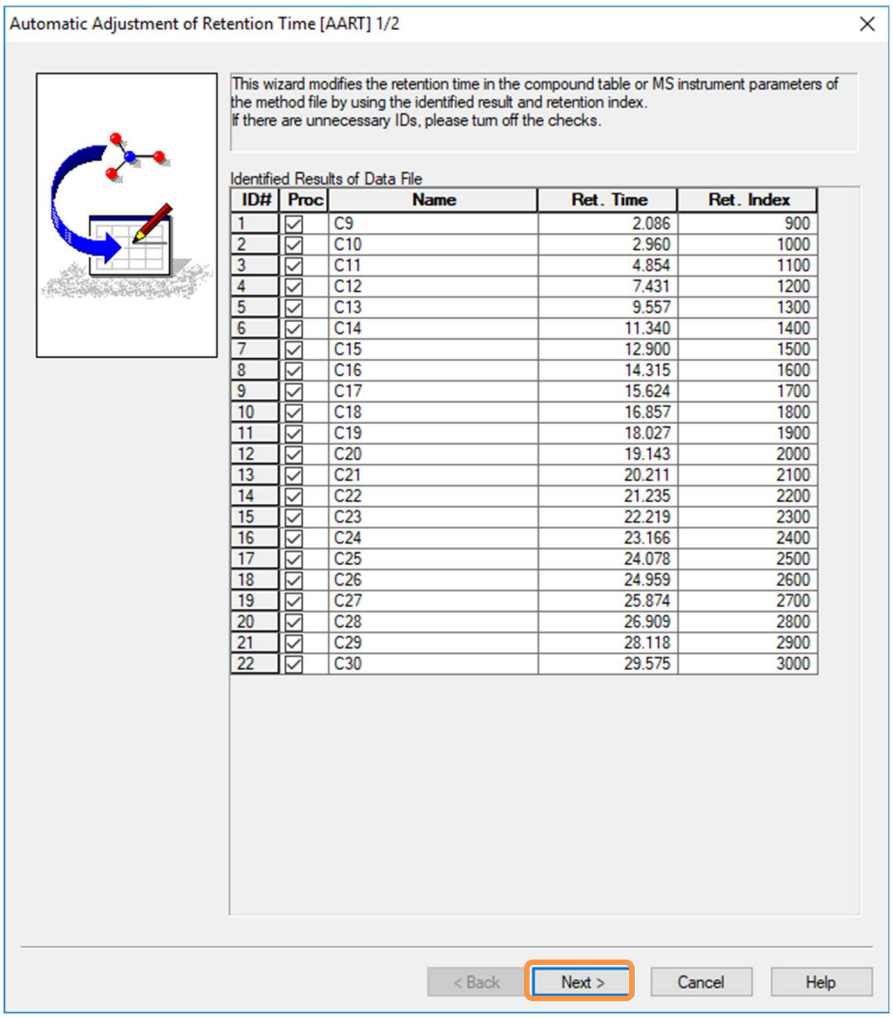
2.1.4 Adjusting Retention Times and Analyzing Samples

1) Click the [AART] icon on the [Compound] assistant bar.

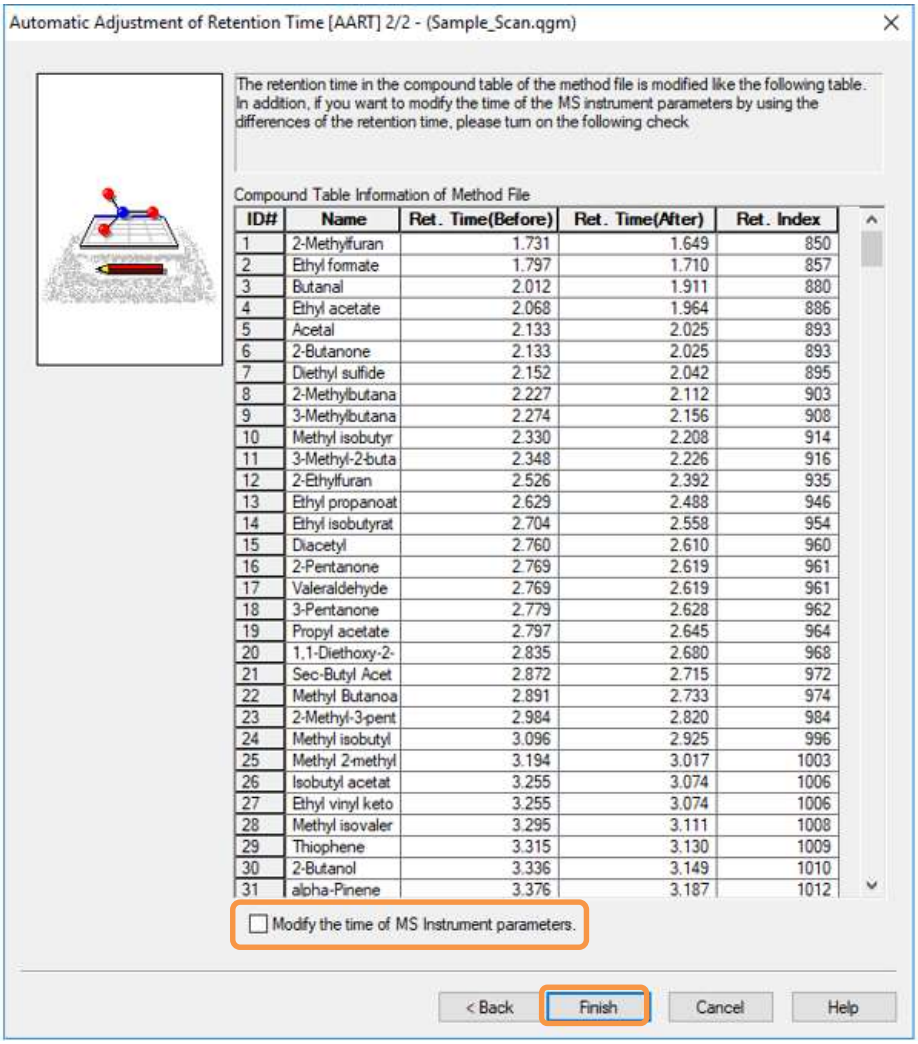
2) Select the method with the retention times to be adjusted and then click [Open].



3) Check the n-alkane identification results and then click [Next]


Automatic Adjustment of Retention Time [AART] 1/2
This wizard modifies the retention time in the compound table or MS instrument parameters of the method file by using the identified result and retention index.
If there are unnecessary IDs, please turn off the checks.
Identified Results of Data File
ID# Proc Name Ret. Time Ret. Index
1 C9 2.086 900
2 C10 2.960 1000
3 C11 4.854 1100
4 C12 7.431 1200
5 C13 9.557 1300
6 C14 11.340 1400
7 C15 12.900 1500
8 C16 14.315 1600
9 C17 15.624 1700
10 C18 16.857 1800
11 C19 18.027 1900
12 C20 19.143 2000
13 C21 20.211 2100
14 C22 21.235 2200
15 C23 22.219 2300
16 C24 23.166 2400
17 C25 24.078 2500
18 C26 24.959 2600
19 C27 25.874 2700
20 C28 26.909 2800
21 C29 28.118 2900
22 C30 29.575 3000
< Back
Next >
Cancel
Help

4) Confirm that the retention times have been adjusted and then click [Finish]. Use the AART function to adjust retention times in the method file.
Clear the [Modify the time of MS Instrument parameters] checkbox and then click [Finish].



NOTE

For scan measurement methods, clear the [Modify the time of MS Instrument parameters] checkbox. Select the checkbox if using this procedure to adjust retention times in a method file created from the database for MRM or SIM measurements. MRM and SIM measurement tables are also automatically adjusted.

5) Then use the method file with adjusted retention times to measure samples.

NOTE

If using an AOC-6000 series or HS-20 series unit, method parameters for the pretreatment system must be specified in the created method file. For a description of that procedure, refer to Appendix 4 Using the Database with an AOC-6000 Series Autosampler or Appendix 6 Using the Database with an HS-20 Series Headspace Sampler.

NOTE

Method files registered in the Smart Aroma Database include a default split ratio of 5. Changing the split ratio setting significantly can cause predicted retention times to deviate from actual retention times, especially for compounds with smaller retention index values. If the values vary significantly, use a standard sample to adjust the retention times based on the retention times predicted from the registered retention index values. (Refer to Appendix 10 Retention Time Adjustment Using a Standard Sample.)

2.1.5 Checking Measurement Results

Measurement results can be analyzed using either the [GCMS Postrun Analysis] program or [Labsolutions Insight GCMS] program.

- GCMS Postrun Analysis: Enables library searches for compounds other than the target compounds, adding compounds, and so on.
- Labsolutions Insight GCMS: Enables data analysis for multiple analytes, which is especially convenient if intending to apply multivariate analysis or other statistical data analysis.

NOTE

Automatic peak detection can sometimes incorrectly identify or not identify peaks in the following cases. Therefore, use the standard spectrum or reference ion ratio registered in the method file or a library similarity search to check measurement results.

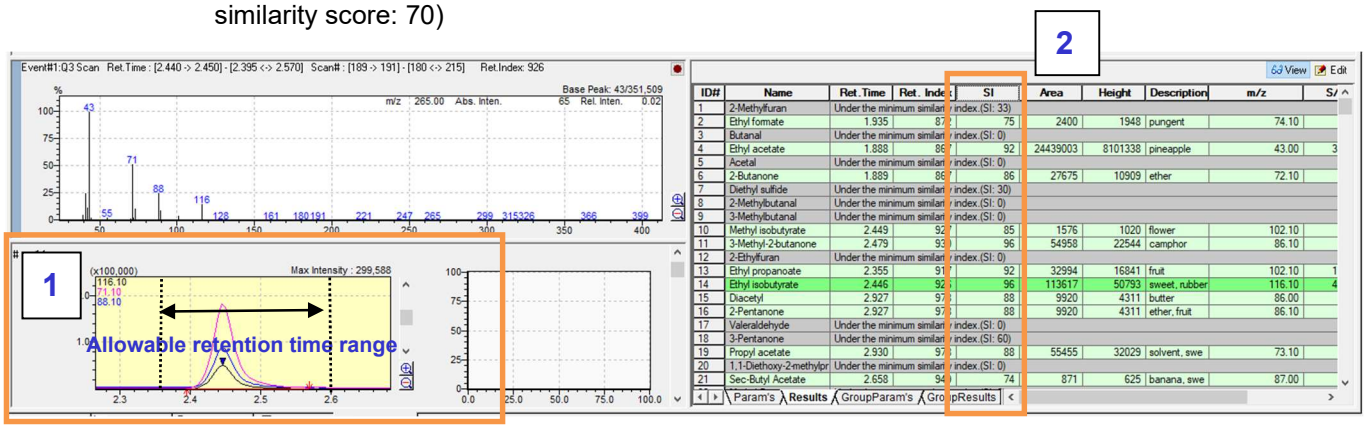
- If the concentration of a target component is extremely low
- If an ion for a target component overlaps with a matrix peak originating from the sample
- If the instrument condition is poor due to contamination of the glass insert, analytical column, or ion source
- If it has been a long time since auto-tuning was performed

> **NOTE**
>
> Retention index values in the database are acquired using liquid injection. Depending on the pretreatment method used, predicted retention times for compounds with smaller retention index values can tend to deviate from actual retention times, due to desorption efficiency or other factors.

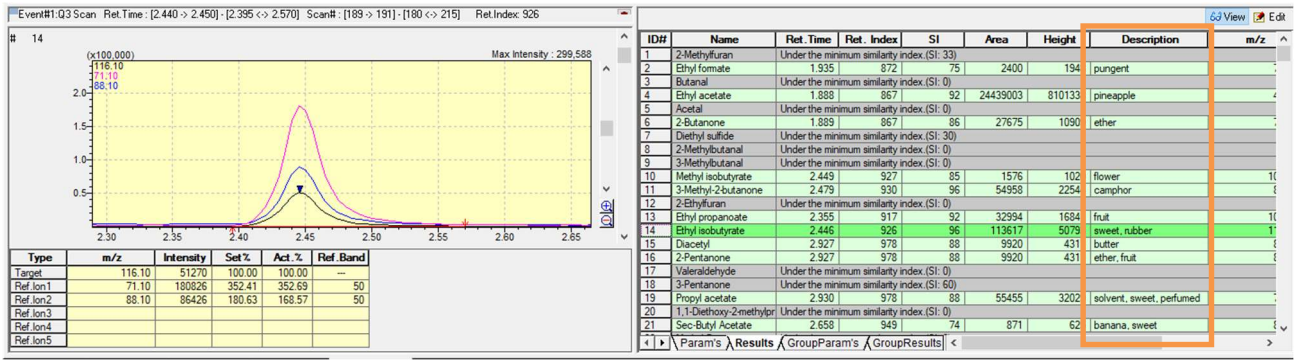
GCMS Postrun Analysis

1) Peak integration and peak identification processes are performed automatically for compounds listed in the compound table, based on the following criteria.

   1 Mass chromatogram peak integration and identification
   *Allowable retention time range: ±0.2 minutes (SH-I-5Sil MS) or 0.3 minutes (SH-PolarWax, InertCap Pure-WAX)
   2 Similarity index (SI) from a reverse search of registered standard spectra (minimum similarity score: 70)



2) Information about the odor characteristics of each compound can be confirmed in the [Description] column of the compound table.

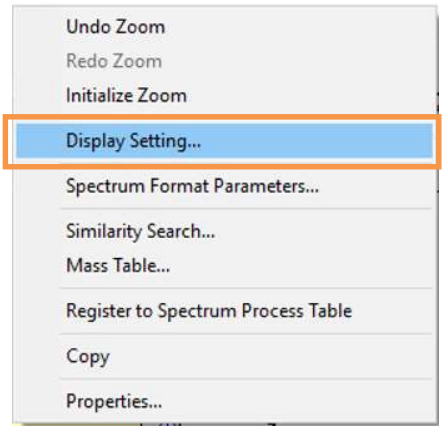


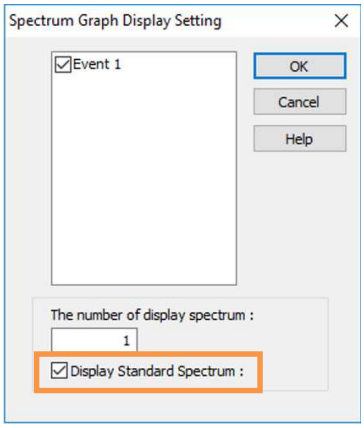
Hint

The compound information registered in the method file includes both mass spectral and reference ion ratio information. Results can be checked more easily by displaying and comparing results to that information.

How to Display Standard Spectra

Right-click in the mass spectrum view and then select [Display Setting…] on the right-click menu and select the [Display Standard Spectrum] checkbox.

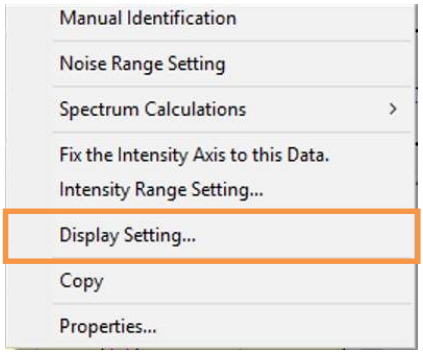


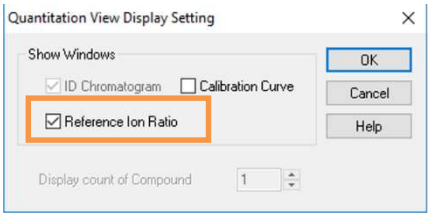


How to Display Reference Ion Ratios

In the compound table, select the compound for the reference ion ratio to be displayed. Then right-click on the mass chromatogram view, click [Display Setting…] on the right-click menu, and select the [Reference Ion Raio] checkbox.

The reference ion ratio information is not used for identifying peaks by automatic peak integration, but it can be used as a reference for checking results.





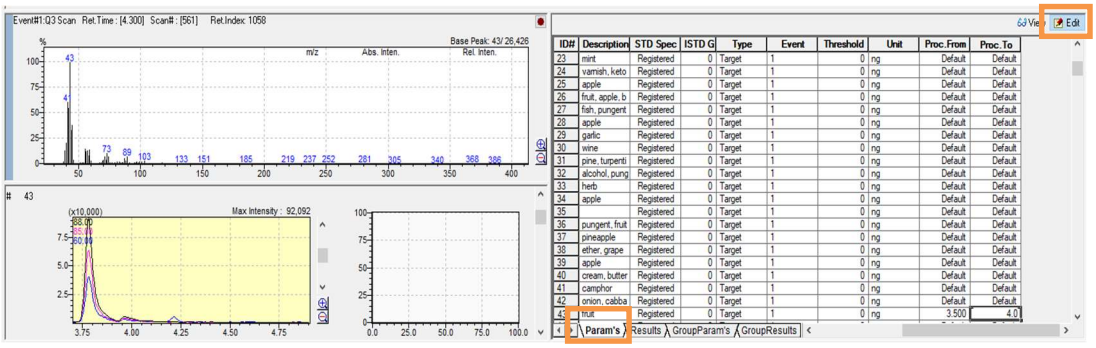
Type	m/z	Intensity	Set %	Act. %	Ref.Band
Target	43.10	1502	100.00	100.00	---
Ref.Ion1	41.10	677	66.00	45.07	50
Ref.Ion2	39.10	206	13.04	13.72	50
Ref.Ion3					
Ref.Ion4					
Ref.Ion5					

NOTE

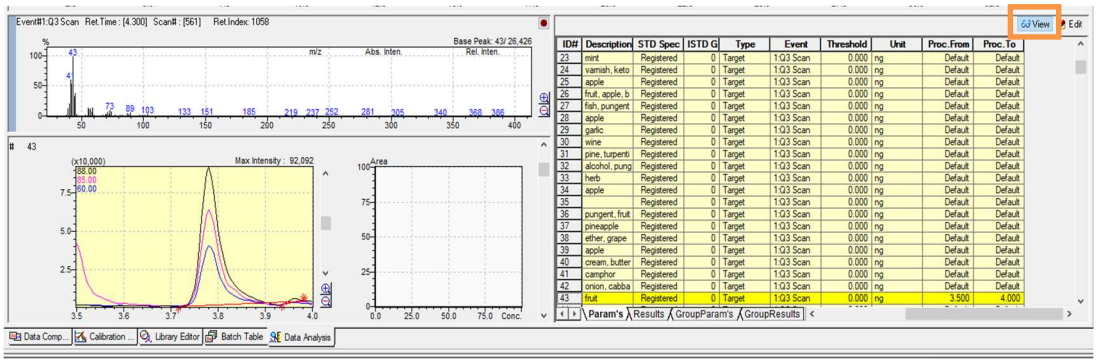
Due to effects from matrix components contained in the sample, retention times for compounds eluted near such peaks can deviate from predicted retention times in some cases.

If the retention time deviation is large enough that retention times extend outside the window, change the window width by the following steps.

1. On the compound table [Param's] tab page, click [Edit].
2. Enter the desired window time settings in the compound table [Proc. From] and [Proc. To] fields. (The default setting is ±1 minutes from the predicted retention time.)



3. Click [View] in the compound table. That changes the window range to the specified duration.



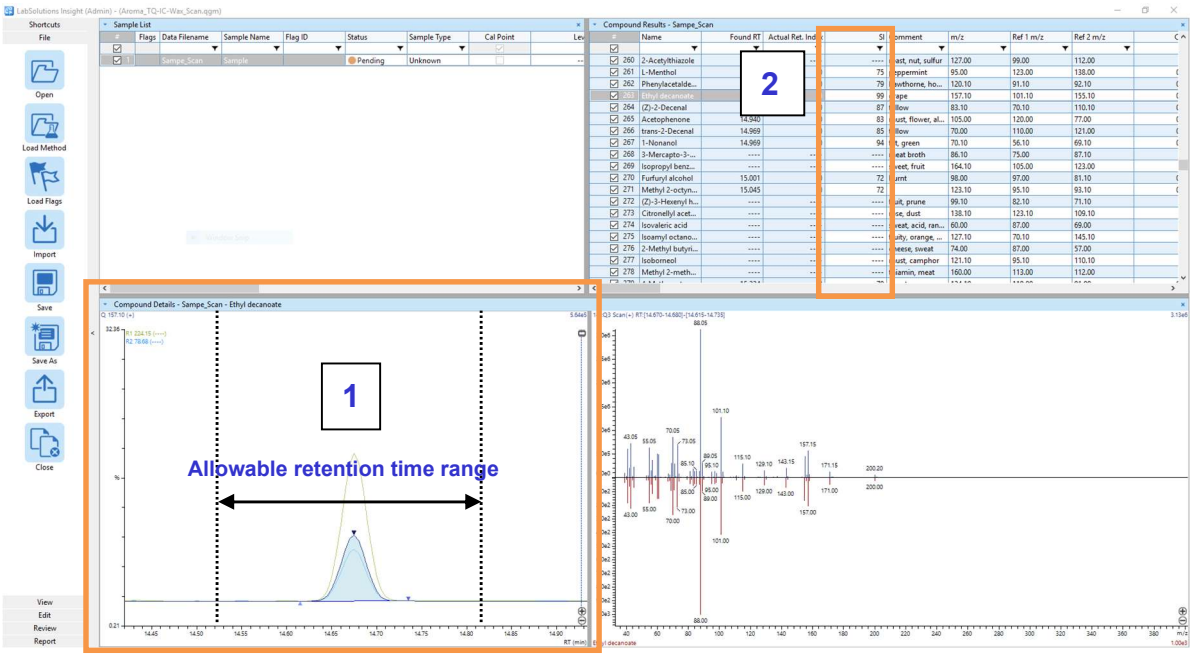
In that case, it is recommended that retention times be adjusted using the retention times for detected peaks, as described in Appendix 10 Retention Time Adjustment Using a Standard Sample.

> **Hint**
>
> For instructions on adding unregistered compounds, refer to Appendix 9 Adding Target Compounds.

Labsolutions Insight GCMS

1) Peak integration and peak identification processes are performed automatically for compounds listed in the compound table, based on the following criteria.

   1 Mass chromatogram peak integration and identification
   *Allowable retention time range: ±0.2 minutes (SH-I-5Sil MS) or 0.3 minutes (SH-PolarWax, InertCap Pure-WAX)
   2 Similarity index (SI) from a reverse search of registered standard spectra (minimum similarity score: 70)

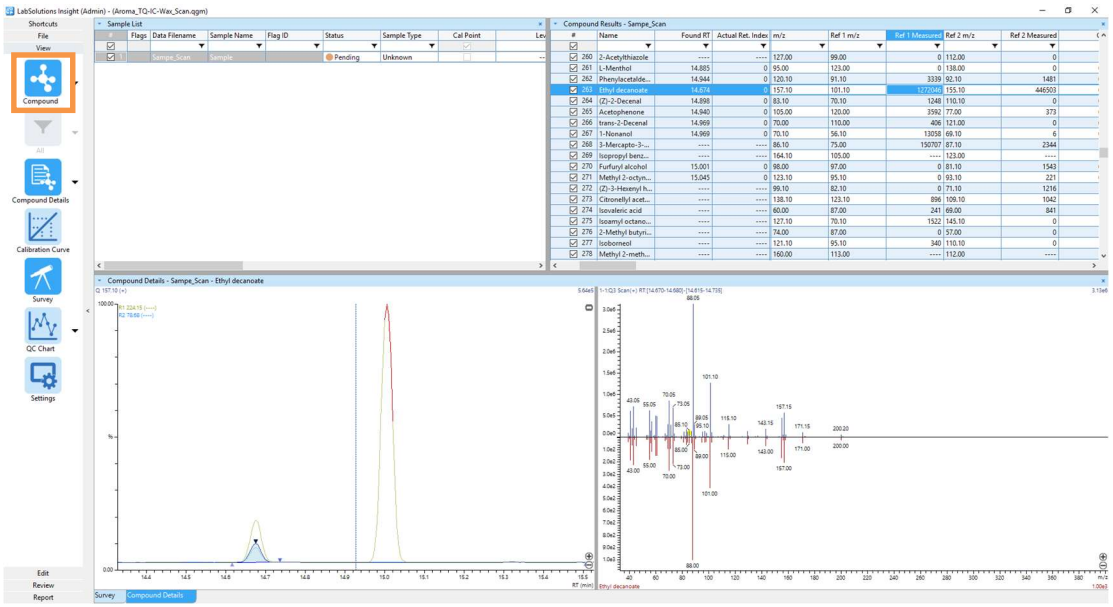


Hint

The compound information registered in the method file includes both mass spectral and reference ion ratio information. Results can be checked more easily by displaying and comparing results to that information.

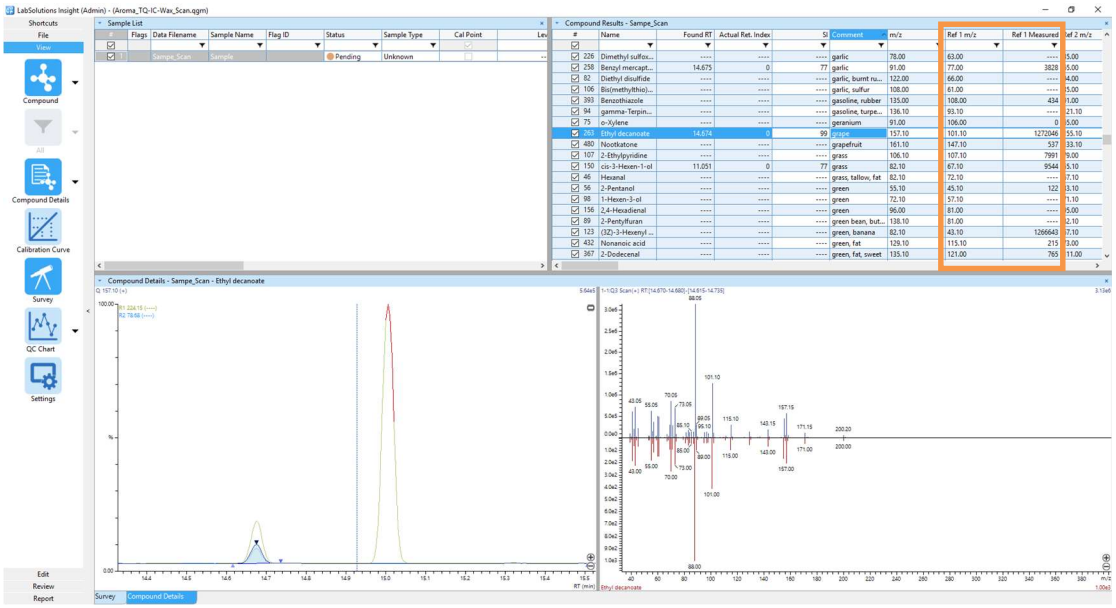
How to Check Standard Spectra

Standard spectra can be checked by displaying the [Compound Details] tab page.

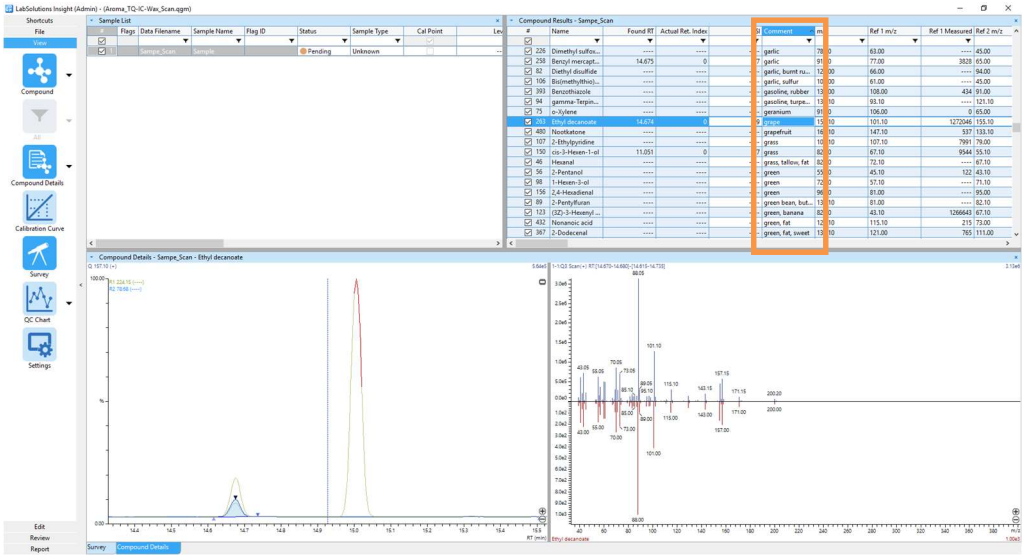


How to Display Reference Ion Ratios

Reference ion ratios can be checked in the [Compound Results] field.



Information about the odor/aroma characteristics of each compound can be confirmed in the [Cmment] column in the [Compound Results] field.



> **Hint**
>
> For instructions on outputting data as a list, so that statistical analysis software can be used to calculate data analysis results, refer to Appendix 11 Outputting Files for Statistical Data Analysis Software.

2.2 Library Operations

2.2.1 Library Search

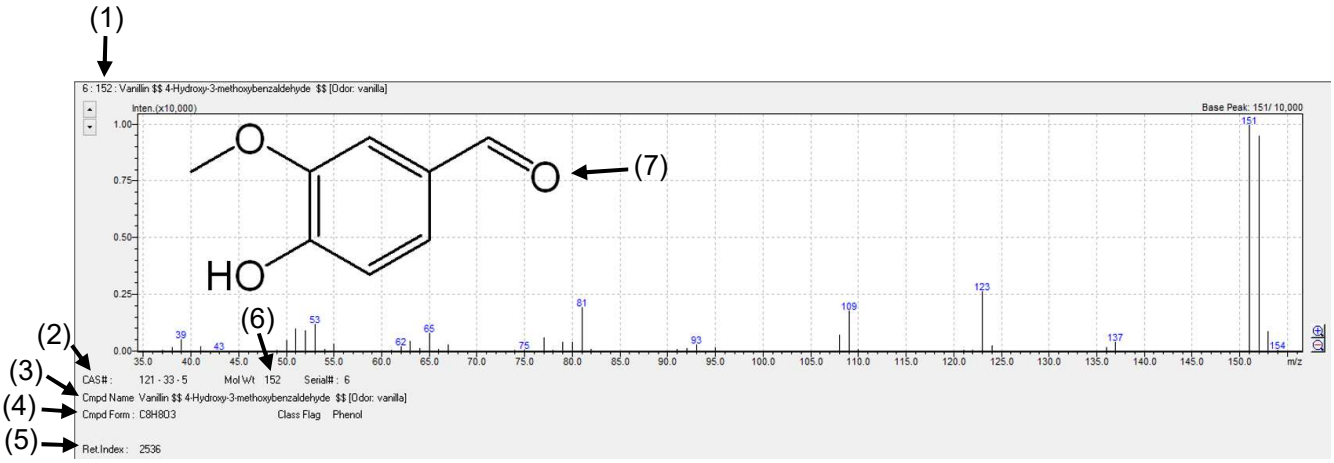
The library can be searched by using it as a private library for Shimadzu GC/MS software. For more details, refer to the instruction manual for the main GC/MS system, which includes instructions for library search operations.

* Normally included in the “C:\GCMSsolution\Manual” folder.

> **NOTE**
>
> The library search functionality in the library is only compatible with the [GCMS Postrun Analysis] program.

2.2.2 Information Registered in the Library

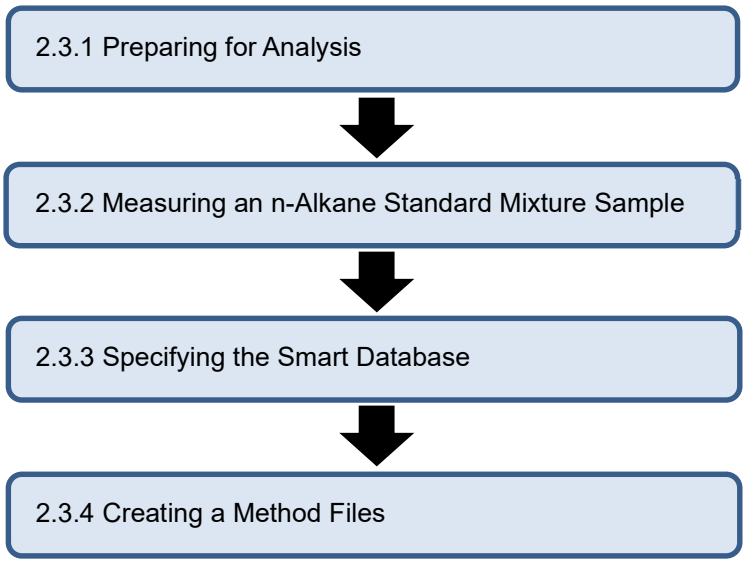
The library includes the following mass spectral information.



(1) Serial numbers registered in the library
(2) Chemical Abstracts Service (CAS) registration numbers
(3) Compound names and corresponding information about the qualitative characteristics of odors/aromas. Odor/aroma characteristics are indicated in square brackets.
(4) Molecular formula
(5) Retention index values calculated for each column using n-alkanes. Data is acquired by the method specified in GC/MS analytical conditions.
(6) Monoisotopic mass values calculated as integer values
(7) Structural formula

2.3 Using a Smart Database to Create Method Files

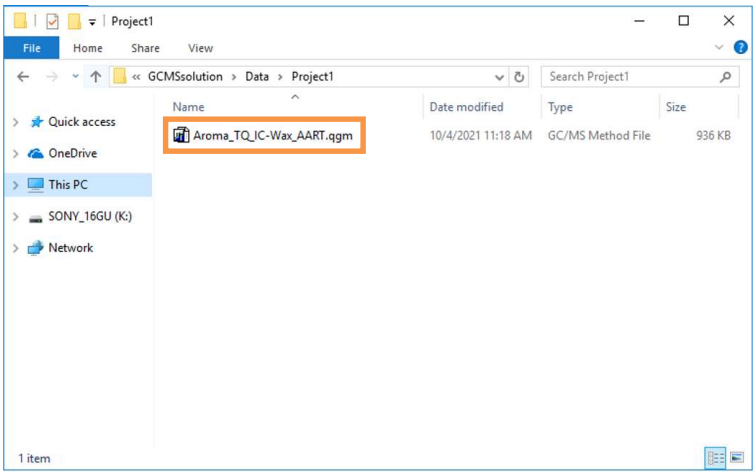
Method files for high-sensitivity targeted analysis using the SIM or MRM mode (or alternatively Scan/SIM or Scan/MRM modes) can be created using a Smart Database.
Widely-targeted scan methods can even be recreated from database files to add a newly registered compound to the method or if the compound table is accidentally edited, for example.
This section describes how to create method files from database files. Method files are created according to the following process flow.



2.3.1 Preparing for Analysis

1) Create a folder for saving respective files (method and data files).
(Example: “C:\GCMSsolution\Data\Project1”)
As an example, the following describes steps for using the Smart Database file “SmartDatabase_Aroma_TQ_IC-PureWAX.xlsm” to create a method file.

2) Copy the necessary n-alkane method file that is stored in the “C:\GCMSsolution\SmartDatabase\Aroma\<column name>” folder and paste it into the folder that was created above.



3) Attach the analytical column. Then specify configuration settings and perform auto-tuning according to the procedure indicated in 2.1.2 Setting Instrument Parameters.

> **NOTE**
>
> In order to use the retention index values registered in the database, use a column with the specified liquid phase type, length, internal diameter, and film thickness made by the specified manufacturer.

2.3.2 Measuring and Identifying n-Alkanes

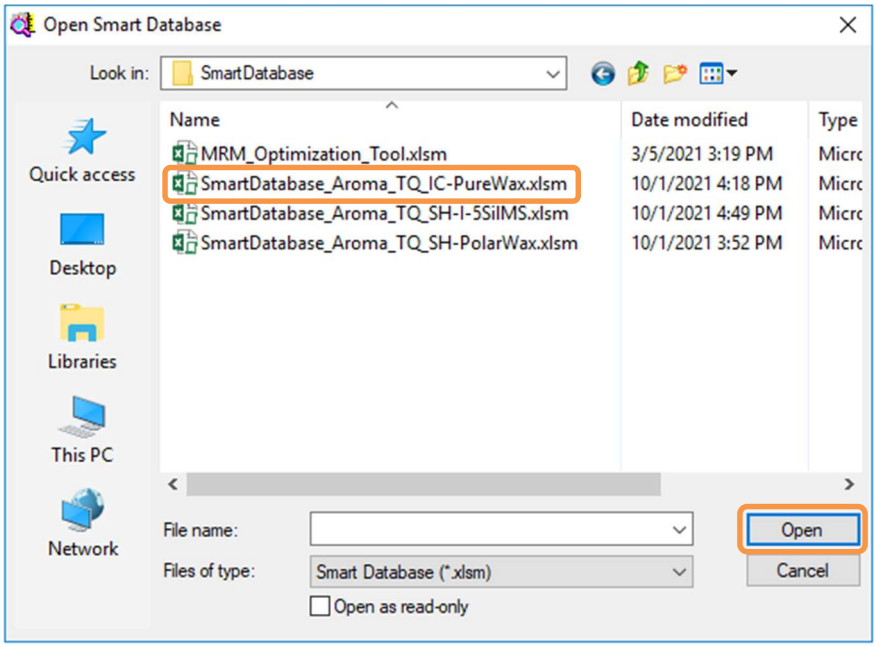
Measure an n-alkane standard mixture solution so retention times registered in the method file can be adjusted. Adjust the registered retention times for target compounds based on the n-alkane identification results and retention index values for the target compounds.
Measure and identify the n-alkanes in the n-alkane standard mixture solution as described in 2.1.2 Measuring an n-Alkane Standard Mixture Sample.

2.3.3 Specifying the Smart Database

1) Click the 

(Smart MRM/SIM) icon on the [Compound] assistant bar.

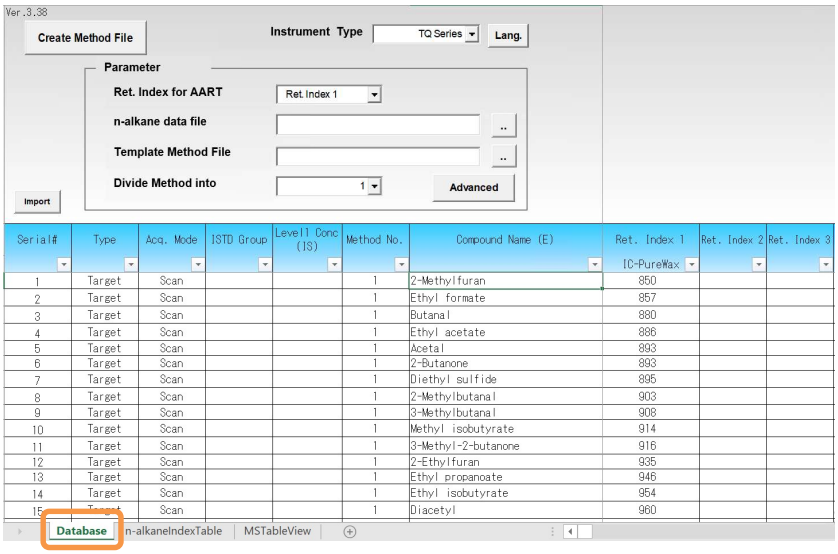
2) Select the Smart Database file (Excel file) based on the column used and then click [Open].



NOTE

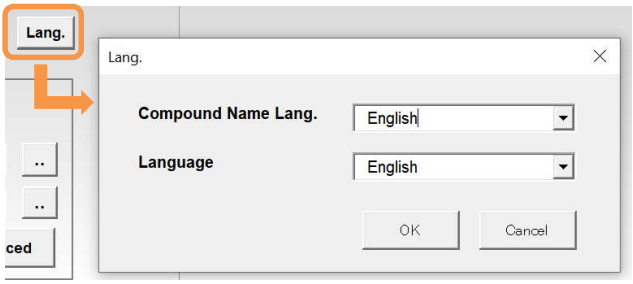
If the following [SECURITY WARNING] message is displayed on the Excel message bar, click [Enable Content].

3) Display the [Database] sheet.



Serial#	Type	Acq. Mode	ISTD Group	Level Conc (IS)	Method No.	Compound Name (E)	Ret. Index 1 IC-PureWax	Ret. Index 2	Ret. Index 3
1	Target	Scan			1	2-Methylfuran	850		
2	Target	Scan			1	Ethyl formate	857		
3	Target	Scan			1	Butanal	880		
4	Target	Scan			1	Ethyl acetate	886		
5	Target	Scan			1	Acetal	893		
6	Target	Scan			1	2-Butanone	893		
7	Target	Scan			1	Diethyl sulfide	895		
8	Target	Scan			1	2-Methylbutanal	903		
9	Target	Scan			1	3-Methylbutanal	908		
10	Target	Scan			1	Methyl isobutyrate	914		
11	Target	Scan			1	3-Methyl-2-butanone	916		
12	Target	Scan			1	2-Ethylfuran	935		
13	Target	Scan			1	Ethyl propanoate	946		
14	Target	Scan			1	Ethyl isobutyrate	954		
15	Target	Scan			1	Diacetyl	960		

4) Click [Lang.] to specify the language for displaying compound names and other text.

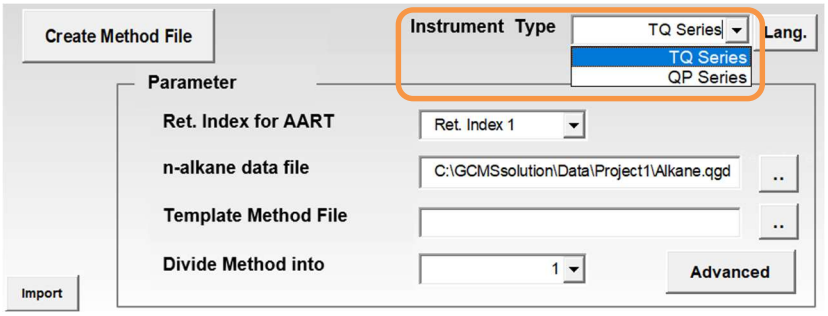


[Compound Name lang.]: The given database only displays compound names in English.
[Language]: Selects the language for displaying information in Smart Database windows.

Hint

The above settings are recorded in the Smart Database.

5) Confirm that the correct instrument type is selected on the [Instrument Type] pull-down menu.



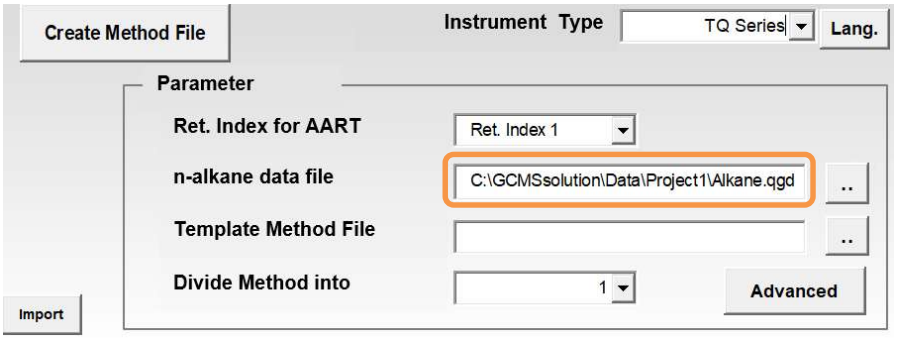
NOTE

Note that an error message will be displayed if the instrument type specified in [Instrument Type] does not match the instrument type specified in configuration settings for data or method files specified in [n-alkane data file] and [Template Method file] fields.

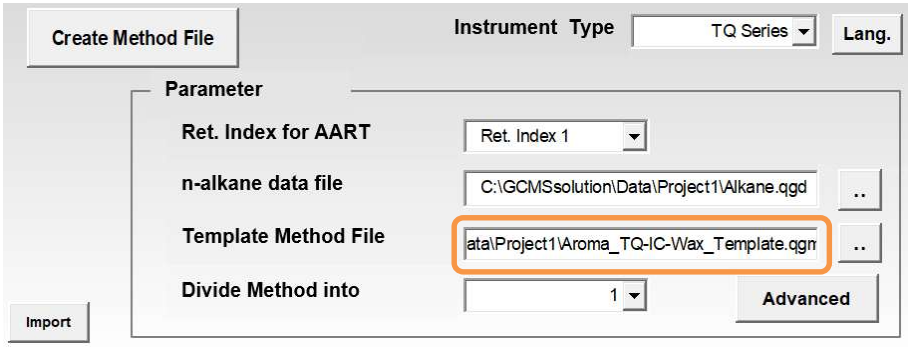
2.3.4 Creating Method Files

2.3.4.1 Setting Target Compounds

1) Confirm that the data file opened in 2.3.2 Measuring and Identifying n-Alkanes is loaded.



2) Of the method files stored in the “C:\GCMSsolution\SmartDatabase\Aroma\<column name>” folder, copy the method file to be used as the template method file to the folder that was created above and specify it in [Template Method File]. The data acquisition and analytical conditions in the method file specified here will be applied when a method is created.



Hint

To create a method file for scan mode analysis, specify the scan method file (“Aroma_QP/TQ_<column name>_Scan.qgm”) in the [Template Method File] field.
To create a method file for SIM or MRM mode analysis, specify the template method file (“Aroma_QP/TQ_<column name>_Template.qgm”) in the [Template Method File] field.

3) In the [Type] field, specify “Target” only for compounds being measured and use the [Delete] key to leave the field blank for other compounds.

Serial#	Type	Acq. Mode	ISTD Group	Level1 Conc (IS)	Method No.	Compound Name (E)	Ret. Index 1 IC-PureWax
1	Target	Scan			1	2-Methylfuran	850
2	Target	Scan			1	Ethyl formate	857
3	Target	Scan			1	Butanal	880
4		Scan			1	Ethyl acetate	886
5	Target	Scan			1	Acetal	893
6	Target	Scan			1	2-Butanone	893
7	Target	Scan			1	Diethyl sulfide	895
8	Target	Scan			1	2-Methylbutanal	903
9	Target	Scan			1	3-Methylbutanal	908
10	Target	Scan			1	Methyl isobutyrate	914
11	Target	Scan			1	3-Methyl-2-butanone	916
12	Target	Scan			1	2-Ethylfuran	935
13	Target	Scan			1	Ethyl propanoate	946
14		Scan			1	Ethyl isobutyrate	954
15	Target	Scan			1	Diacetyl	960
16	Target	Scan			1	2-Pentanone	961

Hint

If retention times are already known for target components from analyzing a standard sample, the method file can be created without using the AART function by using the known retention times.
(Refer to Appendix 10 Retention Time Adjustment Using a Standard Sample.)

4) Specify the measurement mode (select Scan, SIM, or MRM) in the [Acq. Mode] column of the target compound table.

Serial#	Type	Acq. Mode	ISTD Group	Level1 Conc (IS)	Method No.	Compound Name (E)	Ret. Index 1 IC-PureWax
1	Target	Scan			1	2-Methylfuran	850
2	Target	MRM			1	Ethyl formate	857
3	Target	SIM			1	Butanal	880
4		Scan			1	Ethyl acetate	886
5	Target	Scan			1	Acetal	893
6	Target	Scan			1	2-Butanone	893
7	Target	Scan			1	Diethyl sulfide	895
8	Target	Scan			1	2-Methylbutanal	903
9	Target	Scan			1	3-Methylbutanal	908
10	Target	Scan			1	Methyl isobutyrate	914
11	Target	Scan			1	3-Methyl-2-butanone	916
12	Target	Scan			1	2-Ethylfuran	935
13	Target	Scan			1	Ethyl propanoate	946
14		Scan			1	Ethyl isobutyrate	954
15	Target	Scan			1	Diacetyl	960
16	Target	Scan			1	2-Pentanone	961

2.3.4.2 Selecting Ions

In default settings, three ion m/z values are specified for the Scan and SIM modes and two MRM transitions for one compound. To change MRM transitions, check the mass and ion ratio values before selecting the transition.

The ion is used as a target ion if “T” is specified in the [Type] column or as a reference ion if “Ref” is specified. One target ion and up to five reference ions can be specified.

MRM Transition

Ion1				Ion2				Ion3			
Type	m/z	CE	Rati	Type	m/z	CE	Rati	Type	m/z	CE	Rati
T	82.10>54.00	9	100.00	Ref.1	81.10>53.00	6	76.60	Ref.2	82.10>39.00	18	70.48
T	72.10>57.00	3	100.00	Ref.1	72.10>44.10	6	29.54	Ref.2	72.10>43.00	3	23.44
T	70.0>55.0	6	100.00	Ref.1	88.0>61.0	6	14.32	Ref.2	70.0>43.0	18	14.77
T	73.10>45.10	6	100.00	Ref.1	103.10>75.10	6	26.44	Ref.2	103.10>47.10	12	22.51
T	72.10>43.10	6	100.00	Ref.1	72.10>57.10	3	60.62	Ref.2	57.10>41.10	3	1.87
T	90.10>75.10	9	100.00	Ref.1	90.10>62.00	6	91.84	Ref.2	75.00>47.00	6	77.31

m/z for SIM or Scan

Ion1			Ion2			Ion3		
Type	m/z	Rati	Type	m/z	Rati	Type	m/z	Rati
T	82.1	100.00	Ref.1	53.1	70.37	Ref.2	81.1	62.19
T	74.1	100.00	Ref.1	56.1	96.17	Ref.2	73.1	38.55
T	72.1	100.00	Ref.1	44.1	179.52	Ref.2	57.1	58.00
T	43.0	100.00	Ref.1	45.0	15.04	Ref.2	70.0	13.90
T	103.1	100.00	Ref.1	45.1	477.76	Ref.2	73.1	266.85
T	72.1	100.00	Ref.1	43.1	422.88	Ref.2	57.1	33.46
T	90.1	100.00	Ref.1	75.0	87.93	Ref.2	62.0	45.05

1) Check the registered ions or transitions.
2) To change or add an MRM transition, select the setting in the [Type] column pull-down menu.

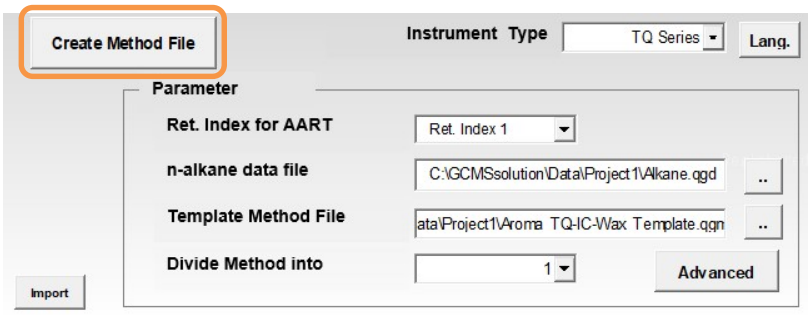
MRM Transition

Ion1				Ion2				Ion3			
Type	m/z	CE	Rati	Type	m/z	CE	Rati	Type	m/z	CE	Rati
T	82.10>54.00	9	100.00	Ref.1	81.10>53.00	6	76.60	Ref.2	82.10>39.00	18	70.48
T	72.10>57.00	3	100.00	Ref.1	72.10>44.10	6	29.54	Ref.2	72.10>43.00	3	23.44
T	70.0>55.0	6	100.00	Ref.1	88.0>61.0	6	14.32	Ref.2	70.0>43.0	18	14.77
T	73.10>45.10	6	100.00	Ref.1	103.10>75.10	6	26.44	Ref.2	103.10>47.10	12	22.51
T	72.10>43.10	6	100.00	Ref.1	72.10>57.10	3	60.62	Ref.2	57.10>41.10	3	1.87
T	90.10>75.10	9	100.00	Ref.1	90.10>62.00	6	91.84	Ref.2	75.00>47.00	6	77.31
T	71.10>43.10	6	100.00	Ref.1	87.10>55.00	9	74.70	Ref.2	102.10>87.10	3	45.62
T	86.10>43.10	9	100.00	Ref.1	86.10>71.10	3	94.06	Ref.2	43.00>41.10	6	42.32
T	96.10>81.00	9	100.00	Ref.1	81.10>53.00	9	85.53	Ref.2	96.10>53.00	21	26.18
T	102.10>74.10	3	100.00	Ref.1	74.10>56.10	6	16.07	Ref.2	74.10>46.00	15	13.42
T	116.10>88.10	3	100.00	Ref.1	88.10>73.10	9	97.45	Ref.2	116.10>73.10	9	64.07
T	86.00>43.00	3	100.00	Ref.1	43.00>15.00	15	47.92	Ref.2	43.00>14.00	30	29.29
T	86.10>71.10	6	100.00	Ref.1	86.10>58.00	3	92.11	Ref.2	86.10>43.10	18	55.28
T	86.10>57.10	6	100.00	Ref.1	86.10>56.00	3	33.92	Ref.2	57.10>42.00	24	5.69
T	61.00>43.10	12	100.00	Ref.1	73.10>43.00	3	82.30	Ref.2	43.10>41.10	3	8.35

2.3.4.3 Creating an MS Table

Specify parameter settings necessary for creating the MS table.

1) Click [Create Method File] to display the [MS Table Parameter] window.



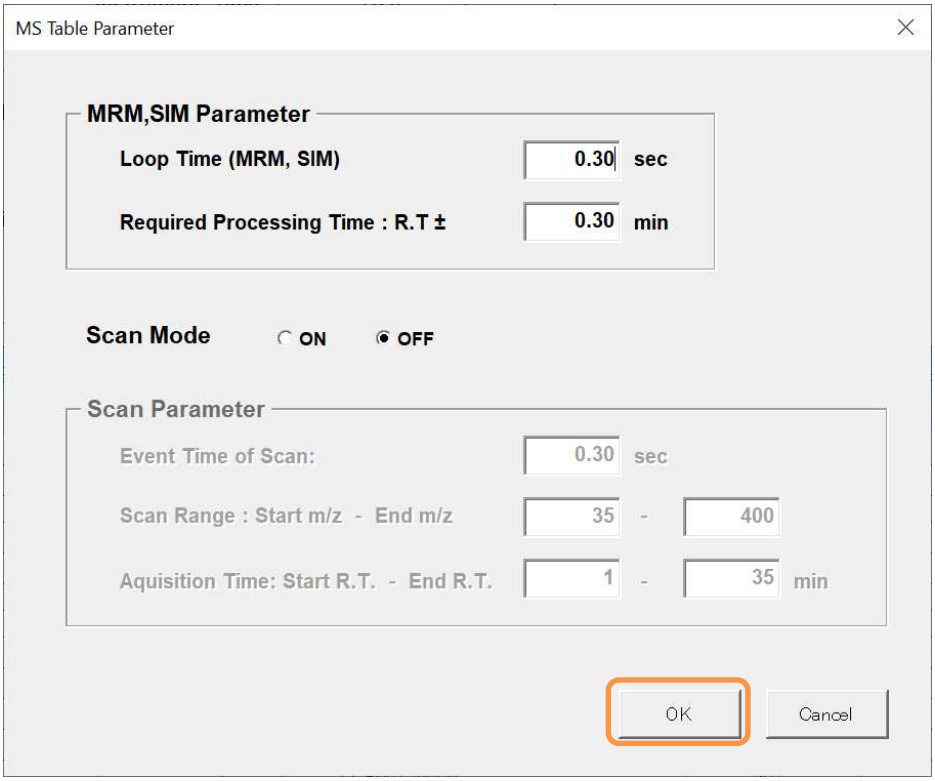
2) Specify [MS Table Parameter] settings.

Creating an MRM or SIM Mode Method File

Configure the settings in the [MRM, SIM Parameter] area. For simultaneous Scan/SIM or Scan/MRM measurements, select [Scan Mode]-[ON] and configure [Scan Mode] settings.

Creating a Scan Mode Method File

Configure the settings in the [Scan Parameter] area.



Hint

Configure the [MS Table Parameter] settings.

[Loop Time (MRM, SIM)] Setting

This indicates the sum of all event times for all compounds specified for a single group. For example, setting it to 0.3 seconds results in acquiring data every 0.3 seconds for each compound. To ensure good reproducibility, at least ten data points must be acquired per peak. However, specifying a short loop time results in lower sensitivity due to less time available for measuring each transition or ion.

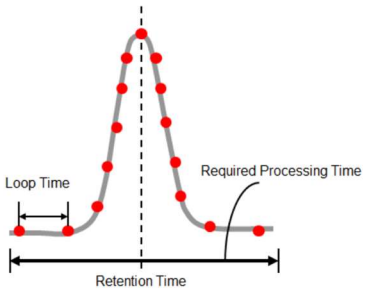
Recommended value: 0.2 to 0.5 seconds

[Required Processing Time] Setting

This indicates the measurement time range, mainly based on the retention times for each compound. For example, setting it to 0.3 minutes specifies acquiring data for each compound within ±0.3 minutes of the specified retention time (0.6 minute time span).

Check retention time deviation by using the method file that was created to measure a sample spiked with a standard sample. Then adjust the setting based on any retention time shift or peak broadening (such as tailing) caused by contaminants or other factors.

Recommended value: 0.3 to 0.5 minutes



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.

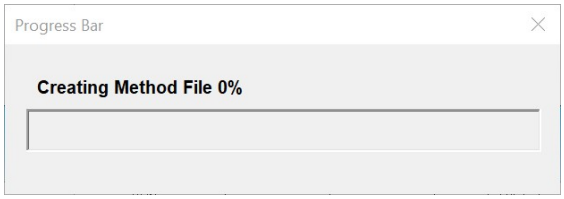
Required Proc. Time for Each Comp. R.T. - X min	Required Proc. Time for Each Comp. R.T. + Y min
0.50	0.50

Hint

The scan parameters for each scan method in this product are indicated below. Use the values as a reference.

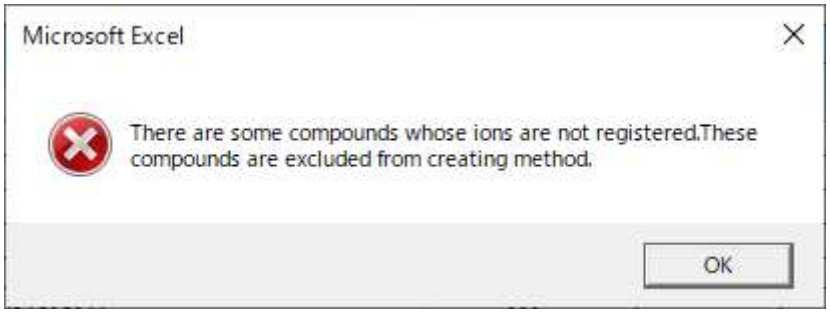
File Name (.qgm)	Event Time (sec)	Scan Range (start-end m/z values)	Analysis Time (min) (start-end R.T. values)
Aroma_QP/TQ_ SH-I-5SilMS _Scan	0.3	35 - 400	1 - 45
Aroma_QP/TQ_ SH-PolarWax_Scan.qgm	0.3	35 - 400	3 - 90
Aroma_QP/TQ_ IC-WAX_Scan.qgm	0.3	35 - 400	1 - 35

3) Clicking [OK] displays the [Progress Bar] window. A method file is created automatically with MS table and compound table configured.



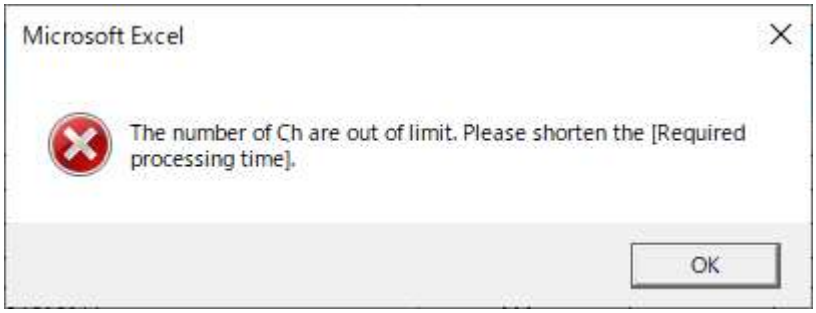
NOTE

If “Target” is specified for a compound without an ion or transition specified, the following type of message is displayed. If [OK] is clicked, an analytical method is created that does not measure compounds without an ion or transition specified.

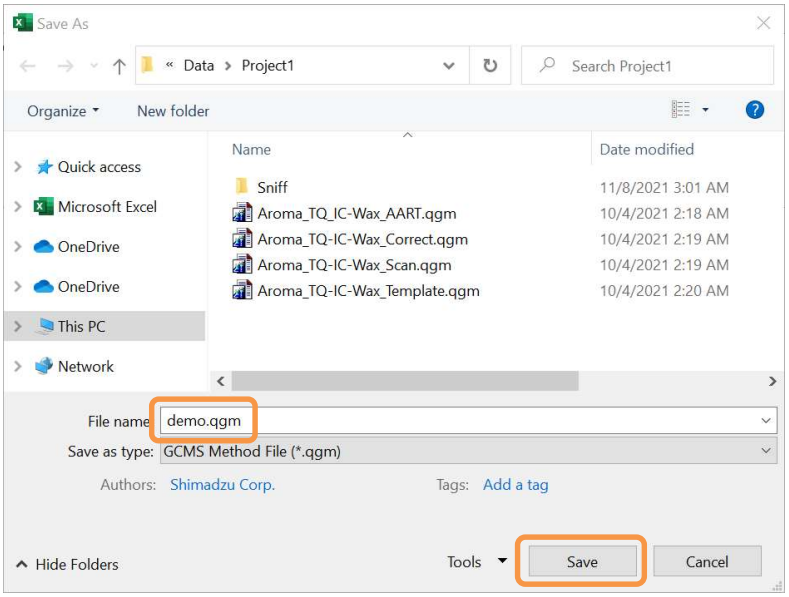


NOTE

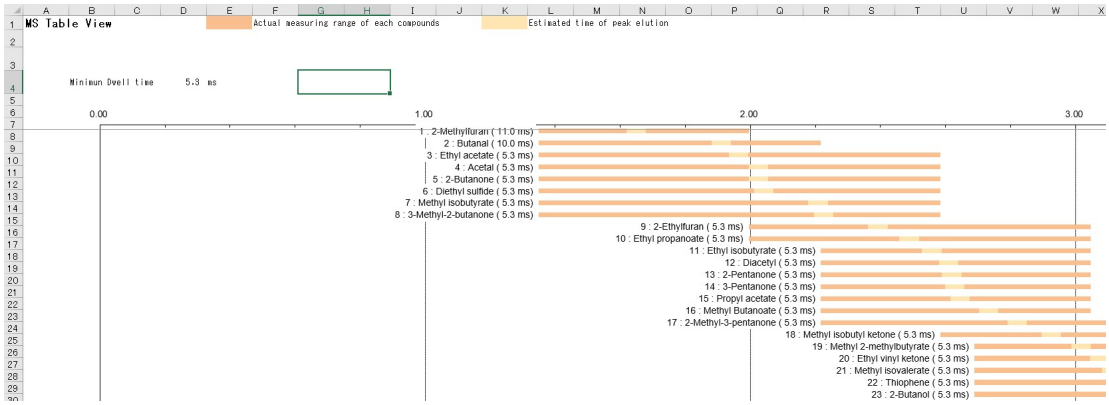
If the number of compounds or number of ions/transitions in the MS table being created exceeds the maximum number of channels, the following type of message is displayed. If that occurs, reduce the number of compounds or the number of ions or transitions for each compound.



4) Name the file as desired and click [Save].

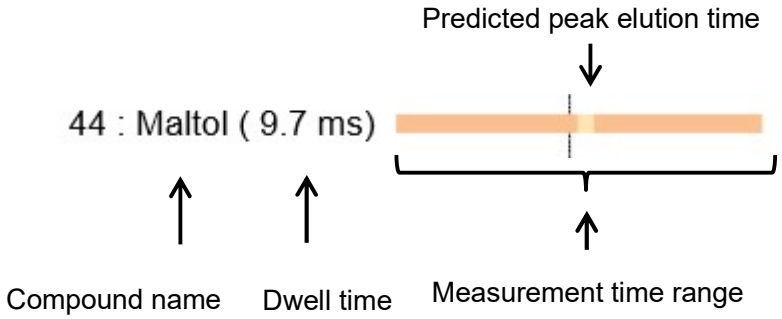


5) Check the time range for MRM and SIM measurements of target compounds. An MS table view is not created for scan mode measurements.



Hint

The measurement time per transition (per ion) (hereafter referred to as “dwell time”) for each target compound specified in the created MRM or SIM analytical method can be confirmed on the [MSTableView] sheet.

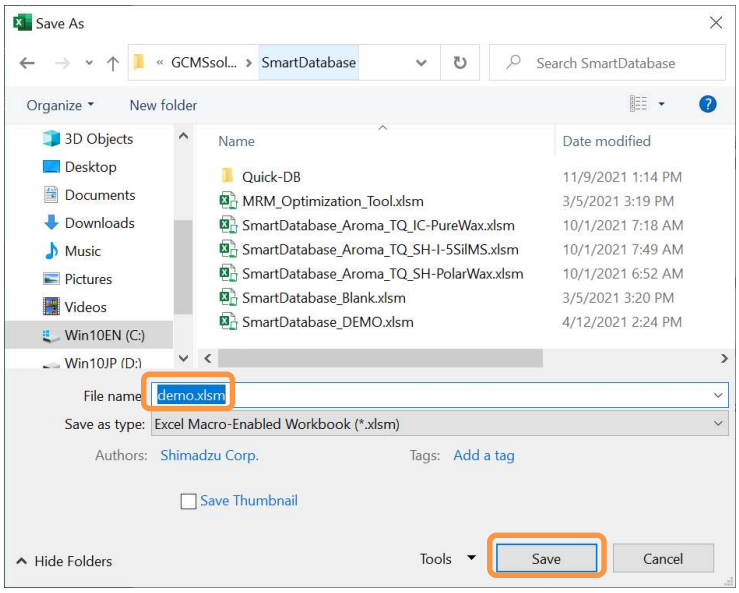


The minimum dwell time value for each target compound can also be confirmed. The more target compounds that are specified, the shorter the dwell time and the lower the sensitivity. It is recommended that settings are specified for the number of compounds and number of transitions/ions that ensure a minimum dwell time of about 2 ms.

NOTE

If using an AOC-6000 series or HS-20 series unit, method parameters for the pretreatment system must be specified in the created method file. For a description of that procedure, refer to Appendix 4 Using the Database with an AOC-6000 Series Autosampler or Appendix 6 Using the Database with an HS-20 Series Headspace Sampler.

6) Name the Smart Database and save it in the folder that was created.



7) Measure samples using the method file created.

2.3.5 Editing Databases

Smart Database files are saved in Excel format, which means they can be freely revised and compounds can be freely added.

If a different column or GC conditions are used, a new method for use under the given conditions can be created by adjusting retention index values. Additional new compounds or transition information can be added by using the MRM Optimization Tool in GCMSsolution to register them.

Revising a Database to Support Different GC Analytical Conditions

Save registered retention index values in an existing database by following the procedure described up to 3.3.3 Registering Retention Index Values in Compound Tables in 3. Registering Additional New Compound Information in a Smart Database in the GCMS Operation Guide – Method Development. Then complete the process by saving the database with a different name.

Registering New Compounds or Ions/Transitions

Register the information by following the series of steps described in 3. Registering Additional New Compound Information in a Smart Database in the GCMS Operation Guide – Method Development. After registering the information, complete the process by saving the database with a different name.

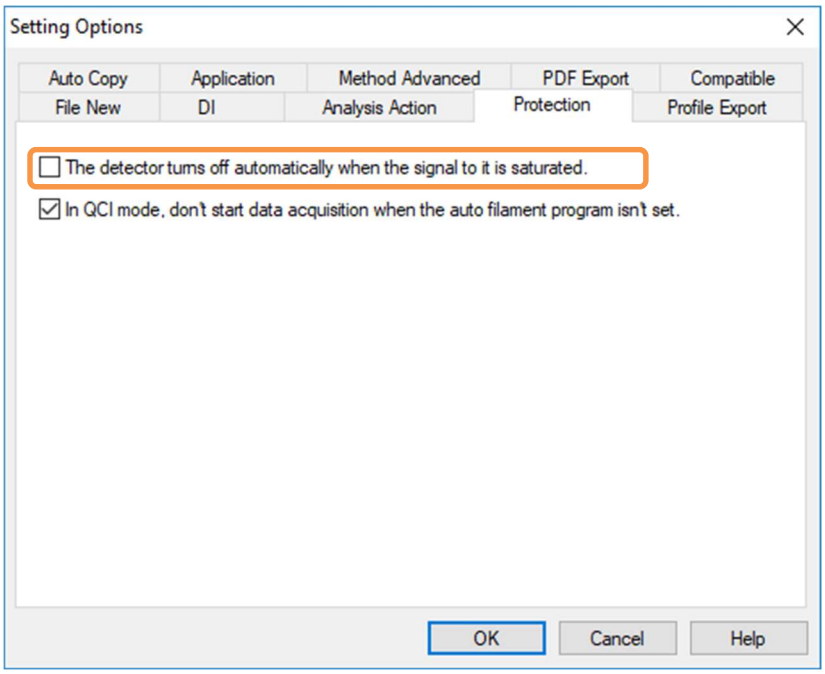
3 Precautions

3.1 Changing Analytical Conditions or Using a Column that was Not Specified

Retention index and calibration curve information registered in the database was obtained by analysis using the analytical conditions in Appendix 1 Analytical Conditions. Beware that changing the analytical conditions or using a column other than the type specified could prevent accurate analysis because it would result in different retention index values.

3.2 Functionality for Protecting the Detector

The detector voltage is specified in advance, but that can cause detector saturation for some samples. If the [The detector turns off automatically when the signal to it is saturated.] checkbox is selected, the filament is automatically switched OFF if detector saturation occurs.
In such cases, that would prevent acquiring data normally, so clear the checkbox for that detector protective function.



Appendix 1 Analytical Conditions

- SH-I-5Sil MS

GC

Injection Mode:	Split (split ratio 5)
Sample Injection Unit Temperature:	250 °C
Oven Temperature:	50 °C (5 min) → (10 °C/min) → 250 °C (10 min)
Carrier Gas:	Helium
Control Mode:	Constant pressure (83.5 kPa)
Purge Flow Rate:	3.0 mL/min

If a Sniffer Unit is Used

Control Mode:	Constant pressure (160 kPa)
Total Flow Rate:	17.2 mL/min
APC1 Pressure:	20 kPa
APC2 Pressure:	200 kPa

MS

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

- SH-PolarWax

GC

Injection Mode:	Split (split ratio 5)
Sample Injection Unit Temperature:	250 °C
Oven Temperature:	40 °C (5 min) → (3 °C/min) → 250 °C (15 min)
Carrier Gas:	Helium
Control Mode:	Constant linear velocity (25.5 cm/s)
Purge Flow Rate:	3.0 mL/min

If a Sniffer Unit is Used

Control Mode:	Constant pressure (230 kPa)
Total Flow Rate:	15.5 mL/min
APC1 Pressure:	20 kPa
APC2 Pressure:	200 kPa

MS

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

- InertCap PureWAX

GC

Injection Mode:	Split (split ratio 5)
Sample Injection Unit Temperature:	250 °C
Oven Temperature:	50 °C (5 min) → (10 °C/min) → 250 °C (10 min)
Carrier Gas:	Helium
Control Mode:	Constant pressure (83.5 kPa)
Purge Flow Rate:	3.0 mL/min

<u>If a Sniffer Unit is Used</u>

Control Mode:	Constant pressure (160 kPa)
Total Flow Rate:	17.2 mL/min
APC1 Pressure:	20 kPa
APC2 Pressure:	200 kPa

MS

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

Appendix 2 List of Customer-Supplied Items

The following reagents and columns are required for creating method files using this product. The customer must provide the following reagents and columns, which are not included with the product.

Reagents

Samples for Retention Time Adjustment

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

Samples for Semi-Quantitative Analysis Correction

- Internal standard/surrogate standard substance mixtures EPA524.1 and EPA524.2 (2000 µg/mL 4-Bromofluorobenzene, 1,2-Dichlorobenzene-d4, Fluorobenzene in methanol) Cat: 47358-U SIGMA-ALDRICH

- Acenaphthene-d10 solution (2000 µg/mL component in methylene dichloride) Cat: 48417 SIGMA-ALDRICH

Columns

SH-I-5Sil MS (30 m x 0.25 mm I.D., df = 0.25 µm) P/N: 221-75954-30
Shimadzu Corp.

SH-PolarWax (60 m x 0.25 mm I.D., df = 0.25 µm) P/N: 227-36247-02
Shimadzu Corp.

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

For information about consumables used in respective pretreatment systems, refer to the instruction manual for the given pretreatment system.

Appendix 3 Calculating Semi-Quantitative Results for Registered Compounds (Scan)

Some compounds registered in the database include registered gradient correction factors based on the internal standard method. Therefore, by measuring the internal standard substance in advance, approximate quantitation values for detected compounds can be calculated based on the area value of the internal standard substance.

NOTE

Applications and Precautions for Using Semi-Quantitative Results

Data measured from injecting 1 µL of a mixture containing a standard sample (10 µg/mL, or 50 µg/mL for some) and an internal standard mixture solution (10 µg/mL) were used to calculate relative response factors (RF)based on the following formula.

$$RF = \frac{Area_{Target} \times Conc_{ISTD}}{Conc_{Target} \times Area_{ISTD}}$$

$Area_{Target}$ and $Conc_{Target}$: Area and concentration values for targets

$Area_{ISTD}$ and $Conc_{ISTD}$: Area and concentration values for internal standards

Of course, that means a different GCMS system or capillary column will result in a different RF value. Therefore, if the RF values from database obtained are used, the resulting values will be different than the true values. Furthermore, the values do not reflect the actual extraction efficiency during pretreatment, which means results can vary significantly depending on the pretreatment system used. However, the values can be used for comparing odor/aroma threshold values (minimum concentration detectable by humans) for detected compounds or as a reference guideline for adjusting standard samples.
Given that calculated concentration values are only approximate, they are less accurate than values determined by conventional quantitative methods based on preparing calibration curves. Therefore, to determine concentration values, calculate quantitative results based on a calibration curve prepared separately.

Hint

The following internal standard samples are recommended.

- EPA 524.2 Fortification Solution 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

<Preparing Samples for internal standard mixture>

Dilute an internal standard/surrogate standard substance mixture (2000 µg/mL) and acenaphthene-d10 solution (2000 µg/mL) with hexane to a concentration of 10 µg/mL.

Preparation Example:

Use a microsyringe to place 50 µL each of the internal standard/surrogate standard substance mixture (2000 µg/mL) and acenaphthene-d10 solution (2000 µg/mL) in a 10 mL volumetric flask and then fill the flask to volume with hexane. Keep the prepared solution stored in a refrigerator or freezer until ready for use.

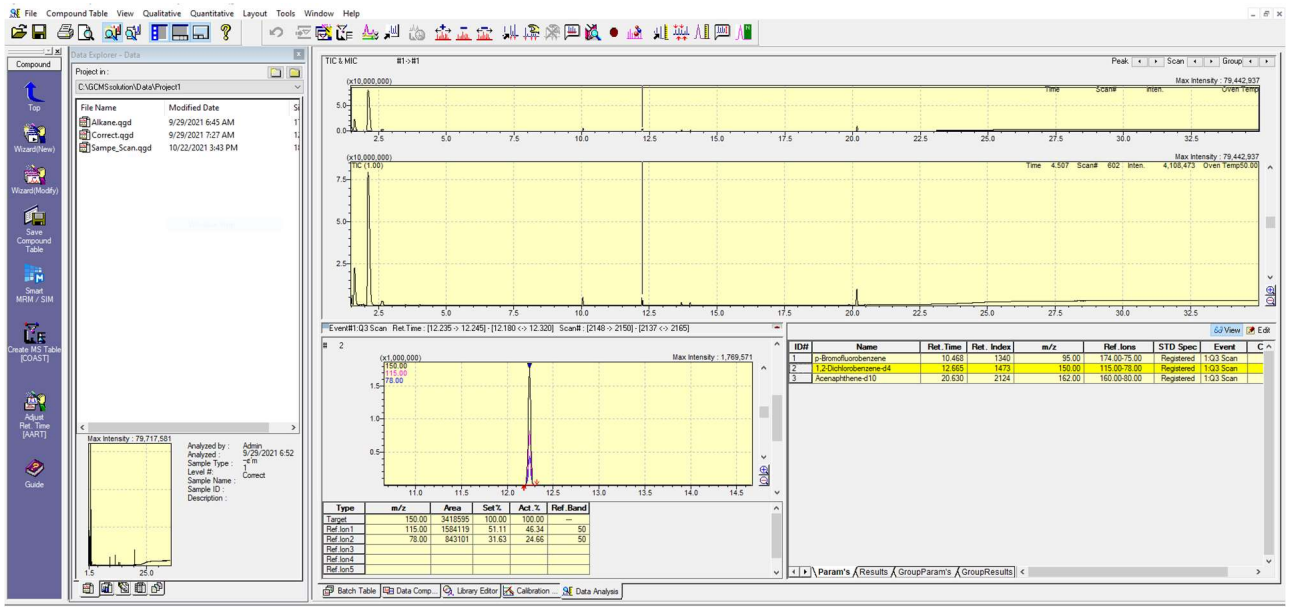
Appendix 3.1 Measuring and Analyzing Standard Samples

n-alkanes are measured for adjusting retention times and internal standard solutions are measured for semi-quantitative analysis.

1) Use a method file for adjusting retention times to analyze the n-alkane solution (50 µg/mL). Use a method file for sensitivity correction to analyze the internal standard mixture solution (10 µg/mL).

2) Load the n-alkane data file and identify the n-alkanes as described in 2.1.2 Measuring an n-Alkane Standard Mixture Sample. After analyzing the data, save the results by overwriting.

3) Load the internal standard data to check and correct the identification results for each component. If any are misidentified, identify them correctly either by manual identification or by manual peak integration. After analyzing the data, save the results by overwriting.



Appendix 3.2 Creating Method Files

1) Load the data file from analyzing n-alkane data.

2) Specify the data as described in 2.3.3 Specifying the Smart Database.

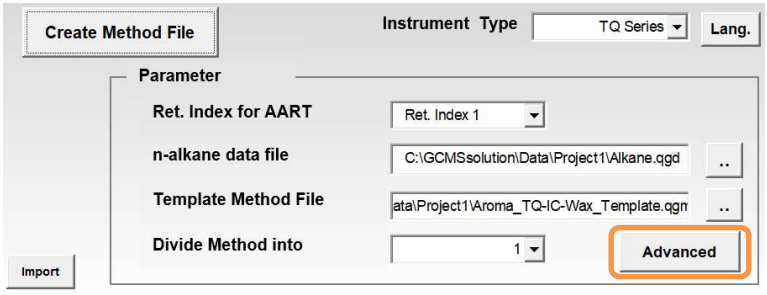
3) Specify measurement target compounds as described in 2.3.4.1 Setting Target Compounds. For the template method file, specify the scan method file (“Aroma_QP/TQ_<column name>_Scan.qgm”).

NOTE

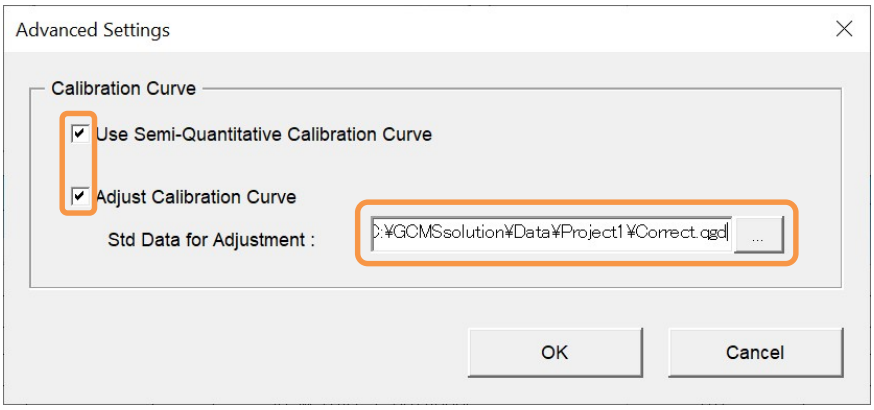
Semi-quantitative analysis is only supported for scan mode analysis.

RF values for registered compounds are calculated based on the area values for target (marked “T”) compounds and ions specified in the database. If target ions are changed, RF values cannot be used for semi-quantitative analysis.

4) Click [Advanced].

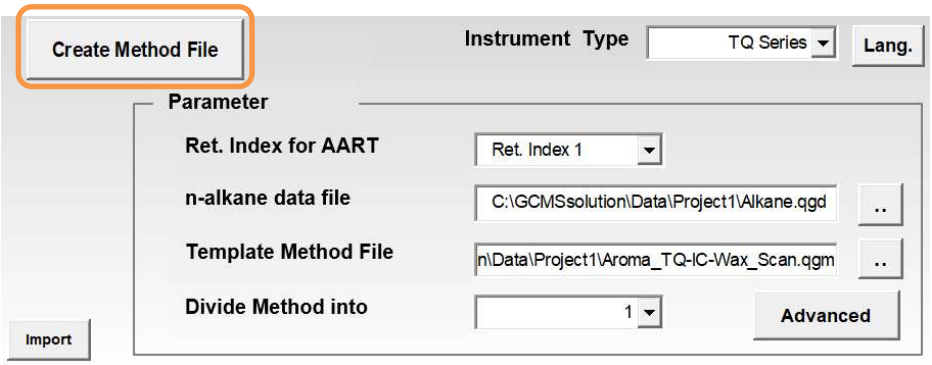


5) Select the [Use Semi-Quantitative Calibration Curve] checkbox.
Select the [Adjust Calibration Curve Std Data for Adjustment:] checkbox and select the data file from the sensitivity correction sample analyzed.

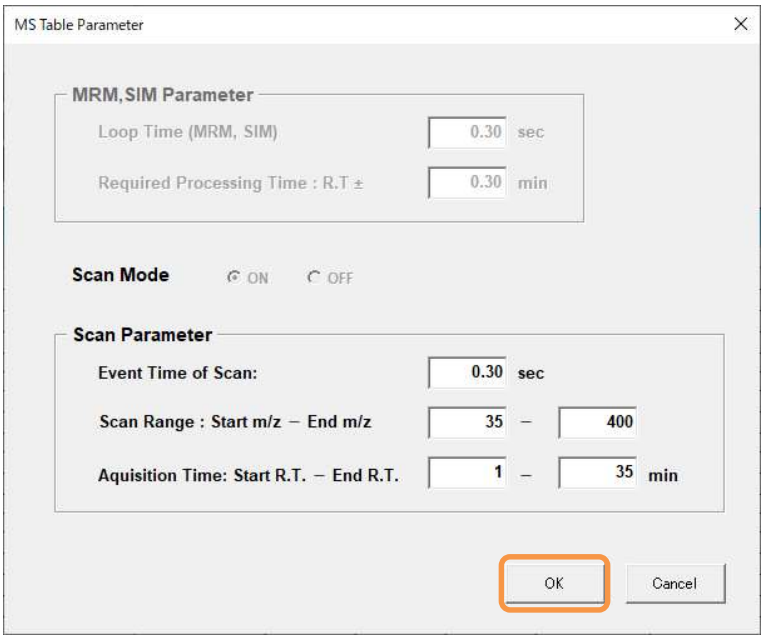


6) Click [OK] to close the window.

7) Click [Create Method File] to display the [MS Table Parameter] window.



8) Click [OK]. Clicking [OK] displays the [Progress Bar] window and automatically creates a method file.



9) Name the file as desired and click [Save].

NOTE

If using an AOC-6000 series or HS-20 series unit, method parameters for the pretreatment system must be specified in the created method file. For a description of that procedure, refer to Appendix 4 Using the Database with an AOC-6000 Series Autosampler or Appendix 6 Using the Database with an HS-20 Series Headspace Sampler.

Appendix 3.3 Analyzing Samples

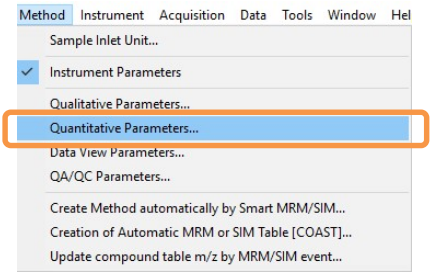
Analyze samples using the method file created for semi-quantitative analysis.

Hint

Semi-quantitative values calculated from analytical results are indicated as absolute values (pg) injected in the GC unit.

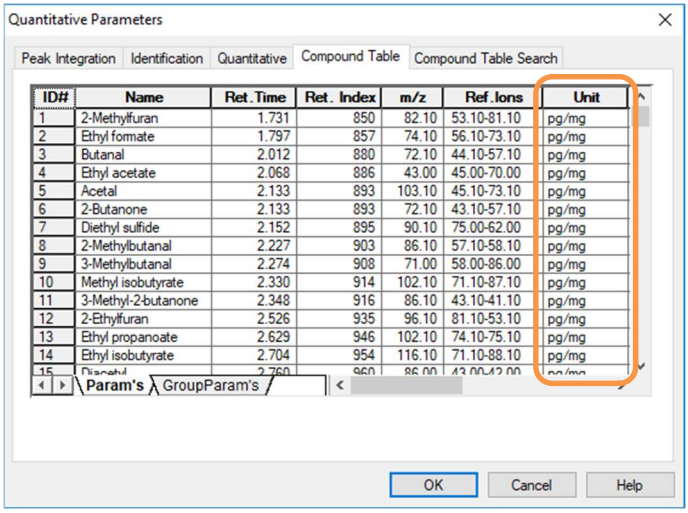
If the sample quantity and dilution factors are entered for batch analysis, the software calculates sample concentrations automatically. Depending on the type of sample, use the following steps to change the concentration units specified in the method file.

1) Load the method file created by the [GCMS Analysis] program.
2) On the [Method] menu bar, click [Quantitative Parameters…].



3) Click the [Compound Table] tab. In the [Unit] column, enter a concentration unit suitable for the weight of the sample obtained.

   Entry example: For solid samples, enter “pg/mg” (sample quantity acquired)
   For liquid samples, enter “pg/µL” (sample quantity acquired)



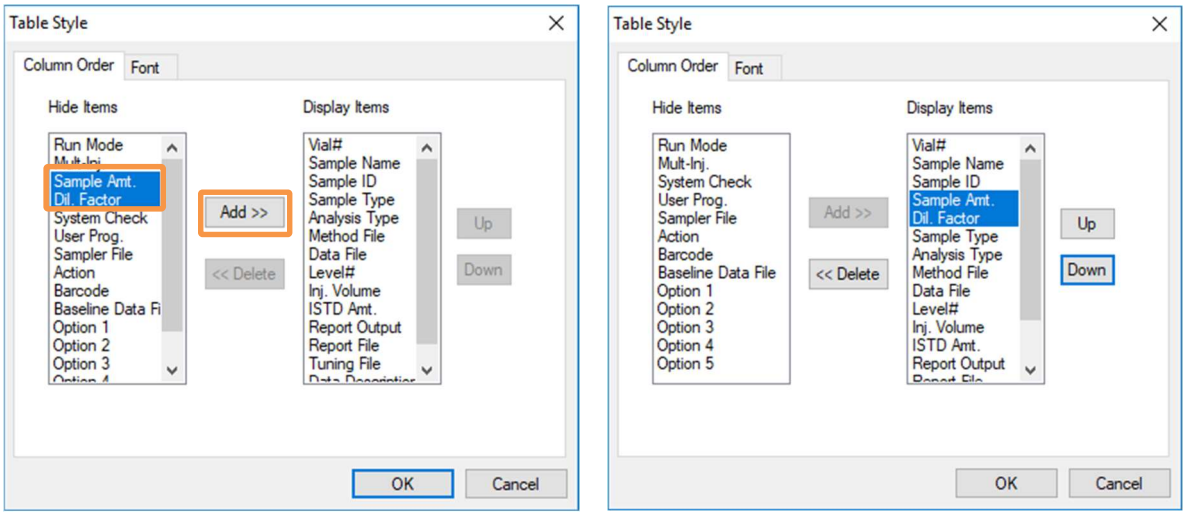
4) Click [OK] to save the method file by overwriting.

1) Place the measurement samples in the autosampler.

2) Open the [Batch Processing] window and create a new batch table.

3) Right-click on the batch table and click [Table Style] on the right-click menu. The [Table Style] window is displayed.

4) In the [Hide Items] field, select [Sample Amt.] and [Dil. Factor]. Then click [Add>>] to add those items to the [Display Items] field.



5) Specify settings in the [Vial #], [Sample Name], and [Sample ID] columns. Specify the created file as the method file. To automatically calculate concentrations in samples based on quantitative results, specify [Sample Amt.] and [Dil. Factor] values.

Folder: C:\Users\shimadzu\Desktop\PIP ENG\SH-1MS

	Vial#	Sample Name	Sample ID	Sample Amt.	Dil. Factor	Sample Type	Analysis Type	Method File
1	1	Sample A	0001	2	100	0:Unknown	IT QT	ax_Scan.qgm
2	1	Sample A	0001	1	1	0:Unknown	IT QT	ax_Scan.qgm
3	2	Sample B	0002	2	100	0:Unknown	IT QT	ax_Scan.qgm
4	2	Sample B	0002	1	1	0:Unknown	IT QT	ax_Scan.qgm
5	3	Sample C	0003	2	100	0:Unknown	IT QT	ax_Scan.qgm
6	3	Sample C	0003	1	1	0:Unknown	IT QT	ax_Scan.qgm
7	4	Sample D	0004	2	100	0:Unknown	IT QT	ax_Scan.qgm
8	4	Sample D	0004	1	1	0:Unknown	IT QT	ax_Scan.qgm
9	5	Sample E	0005	2	100	0:Unknown	IT QT	ax_Scan.qgm
10	5	Sample E	0005	1	1	0:Unknown	IT QT	ax_Scan.qgm
11	6	Sample F	0006	2	100	0:Unknown	IT QT	ax_Scan.qgm
12	6	Sample F	0006	1	1	0:Unknown	IT QT	ax_Scan.qgm
13	7	Sample G	0007	2	100	0:Unknown	IT QT	ax_Scan.qgm
14	7	Sample G	0007	1	1	0:Unknown	IT QT	ax_Scan.qgm
15	8	Sample H	0008	2	100	0:Unknown	IT QT	ax_Scan.qgm
16	8	Sample H	0008	1	1	0:Unknown	IT QT	ax_Scan.qgm

Hint

Examples of [Sample Amt.] and [Dil. Factor] Settings

The [Sample Amt.] value is the eventual quantitation value calculated by dividing the concentration value determined from the calibration curve by the value specified here. The [Dil. Factor] value is the eventual quantitation value calculated by multiplying the concentration value determined from the calibration curve by the value specified here.

[Sample Amt.]

Enter the acquired sample weight in the compound table, in terms of the [Unit] setting.
Example: Enter “2.5” for a sample weighing 2.5 mg.

[Dil. Factor]

Mainly entered for solvent extracted samples injected as a liquid, enter values as a ratio of the final extract solution divided by the sample injection volume.

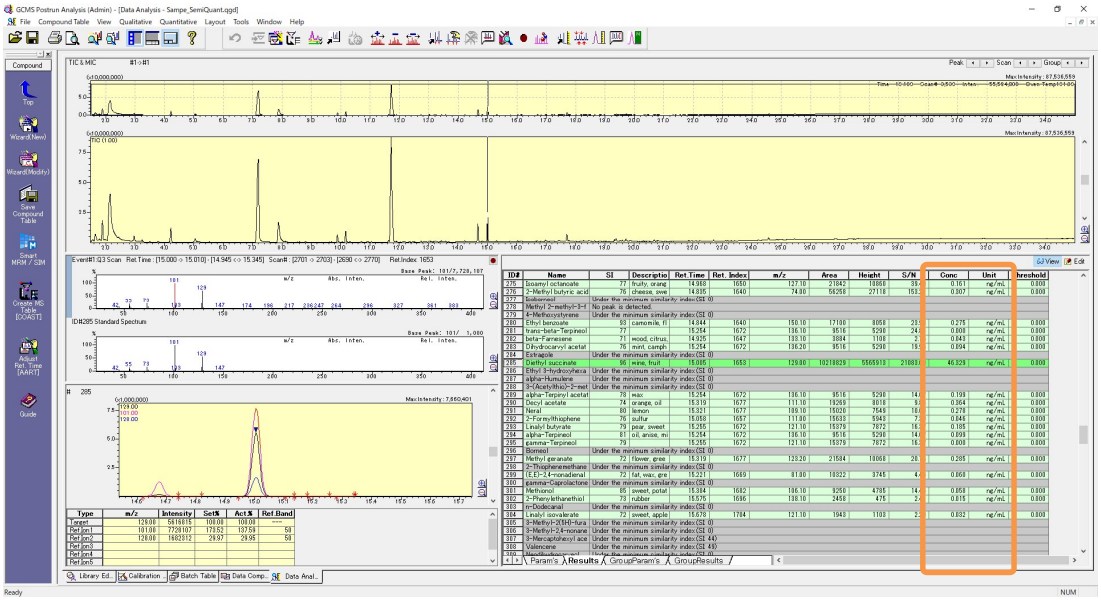
Example: Assume a 2.5 mg solvent extracted sample is used to prepare 1000 µL of a final extract solution, from which 1 µL is injected into the GC/MS system.
Enter Sample Amt.: "2.5" (mg) and Dil Factor: 1000 (µL)/ 1 (µL) = "1000".

6) Name and save the batch file.

7) Execute realtime batch analysis.

Appendix 3.4 Checking Measurement Results

1) Check the identification results. The quantitative results are displayed for identified compounds.



Appendix 4 Using the Database with AOC-6000 Series Autosampler

This chapter describes the operating procedure when using an AOC-6000 series sampler as a pretreatment system. Using an AOC-6000 series sampler requires that AOC-6000 method parameters consistent with the given sample injection method are specified in the methods file.

> **Hint**
>
> For AOC-6000 series operating instructions, refer to the AOC-6000 Operation Guide and AOC-6000 Control Software Instruction Manual.

Appendix 4.1 Instrument Configuration Settings

Configure instrument configuration settings as described in 3.2 Setting the Configuration in the AOC-6000 Control Software Instruction Manual.

Appendix 4.2 Analyzing n-Alkanes

If using an AOC-6000 series system to analyze n-alkanes for retention time adjustment, use the liquid injection method.

1) Install a liquid injection syringe adapter (10 µL capacity) on the AOC-6000 series system. For adapter installation instructions, refer to 4. PAL Tool in the AOC-6000 Operation Guide.

2) Specify method parameter settings for the n-alkane measurement method (“Aroma_QP/TQ_<column>_AART.qgm”) as described in 4. Method Settings in the AOC-6000 Control Software Instruction Manual.
Select “Liquid Injection” for the AOC-6000 method and specify liquid injection parameter settings indicated in Appendix 4.4.

3) Place an n-alkane standard mixture sample (50 µg/mL) on the AOC-6000 rack.

4) Analyze the n-alkane sample as described in 5. Measuring Samples in the AOC-6000 Control Software Instruction Manual.
Set the injection volume to 1 µL.

Hint

If an AOC-6000 series system is used for semi-quantitative analysis according to the procedure in Appendix 3 Calculating Semi-Quantitative Results for Registered Compounds, then also analyze the internal standard substance using the same liquid injection method as used for the n-alkanes. However, note that semi-quantitative results do not reflect extraction efficiency during pretreatment, so they can differ significantly from actual quantitation values, depending on the sample injection method used.

Appendix 4.3 Analyzing Samples

Revise the method parameter settings based on the sample injection method used.

1) Use the n-alkane measurement data to adjust the retention times in the method file as described in 2.1.4 Adjusting Retention Times and Analyzing Samples. If using a database to create the method, follow the procedure in 2.3 Using a Smart Database to Create Method Files, starting from 2.3.3 Specifying the Smart Database.

2) Replace the GC injection port insert liner based on the sample injection method used for sample analysis. Also install a syringe tool for the AOC-6000 system.

3) In the method adjusted or created in step 1), specify method parameter settings for the sample injection method used, according to the procedure described in 4. Method Settings in the AOC-6000 Control Software Instruction Manual. For information about AOC-6000 method parameter settings, refer to Appendix 4.4 Examples of Method Parameter Settings.

4) Place the measurement samples in the AOC-6000 rack and start analyzing them using the method file indicated in step 3).

Appendix 4.4 Examples of Method Parameter Settings

Use the following examples as a reference for specifying method parameter settings for respective AOC-6000 sample injection methods. For more information about each parameter, refer to Appendix – in the AOC-6000 Control Software Instruction Manual.

- **Liquid Injection**

  Analysis Group

Syringe Tool:	Selected from the list of syringe tools for liquid injection (such as LS1 or LS2).
Pre Wash Cycles:	3
Sample Rinse Cycles:	1

Sample Aspirate Flow Rate:	1 µL/s
Sample Post Aspirate Delay:	2 s
Injector:	Selected from the list of injectors (such as Injector1 or Injector2).
Injection Flow Rate:	50 µL/min
Post Wash Cycles:	3

Setup Group

Gas Chromatograph:	Selected from the list of GC units (such as GC1).
Cooled Stack 1:	Not selected.
Cooled Stack 1 Temperature:	20 °C
Cooled Stack 2:	Not selected.
Cooled Stack 2 Temperature:	20 °C
Pre Wash Station:	Selected from the list of wash stations.
Post Wash Station:	Selected from the list of wash stations.
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:	Disable

Advanced Group

Pre Wash Solvent Volume:	8 µL
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:	1 µL/s
Sample Vial Penetration Speed:	50 mm/s
Sample Rinse Volume:	3 µL
Filling Strokes Count:	5
Filling Strokes Volume:	3 µL
Filling Strokes Aspirate Flow Rate:	5 µL/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 µL
Injector Penetration Speed:	100 mm/s

Pre Injection Dwell Time:	0 s
Post Injection Dwell Time:	0.3 s
Post Wash Solvent Volume:	8 µL
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:	1 µL/s

NOTE

The settings above are for using a syringe with a metal plunger. If a syringe with a PTFE plunger is used, change the setting for [Filling Stroke Aspirate Flow Rate] from 5 µL/s to 2 µL/s, for example, as necessary to ensure samples are aspirated slowly enough to prevent bubbles inside the syringe.

- **SPME Injection**

Analysis Group

Fiber Tool:	Selected from the list of syringe tools for SPME.
Conditioning Port:	Selected from the pull-down menu.
Conditioning Temperature:	Set to the recommended value for the selected SPME fiber.
Pre Conditioning Time:	Set to the recommended value for the selected SPME fiber.
Incubation Temperature:	80 °C
Incubation Time:	5 min
Agitator Speed:	250 rpm
Sample Vial Depth:	22 mm
Sample Extract Time:	30 min
Injector:	Selected from the list of injectors (such as Injector1 or Injector2).
Sample Desorb Time:	2 min
Post Conditioning Time:	Set to the recommended value for the selected SPME fiber.
Analysis Time:	Sum of GC program time, cooling time, and equilibration time, plus an extra margin.

Setup Group

Gas Chromatograph:	Selected from the list of GC units (such as GC1).
Sync Before Extraction End Time:	0 min
Agitator:	Selected from the list of agitators.
Do Agitation:	True
Heat Agitator:	True
Wait For Readiness Agitator:	True
Internal Standard Station:	Not selected.
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

NOTE

Select the type of liquid phase for the SPME fiber based on the targeted components.

- **SPME Arrow Injection**

Analysis Group

SPME Arrow Tool:	Selected from the list of syringe tools for SPME Arrow.
Conditioning Port:	Selected from the pull-down menu.
Conditioning Temperature:	Set to the recommended value for the selected SPME Arrow.
Pre Conditioning Time:	Set to the recommended value for the selected SPME Arrow.
Incubation Temperature:	80 °C
Incubation Time:	5 min
Agitator Speed:	250 rpm
Stirrer Speed:	250 rpm
Sample Vial Depth:	33 mm

Sample Extract Time:	30 min
Injector:	Selected from the list of injectors (such as Injector1 or Injector2).
Sample Desorb Time:	2 min
Post Conditioning Time:	Set to the recommended value for the selected SPME Arrow.
Analysis Time:	Sum of GC program time, cooling time, and equilibration time, plus an extra margin.

Setup Group

Gas Chromatograph:	Selected from the list of GC units (such as GC1).
Wash Station:	1
Do Pre Wash:	False
Agitator:	Selected from the list of agitators.
Stirrer:	True
Do Agitation:	True
Heat Agitator and Stirrer:	True
Internal Standard Station:	Not selected.
Internal Standard Position:	1
Injector Penetration Depth:	54 mm
Injection Signal Mode:	Before Expose
Do Post Wash:	False

Advanced Group

Pre Solvent Index:	1
Pre Wash Time:	0 min
Pre Wash Vial Depth:	35 mm
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
Post Solvent Index:	1
Post Wash Time:	0 min
Post Wash Vial Depth:	35 mm

NOTE

Select the type of liquid phase for the SPME Arrow based on the targeted components.

- **Headspace Injection**

Analysis Group

Syringe Tool:	Selected from the list of syringe tools for HS units (such as HS1 or HS2).
Incubation Temperature:	60 °C
Incubation Time:	30 min
Syringe Temperature:	80 °C
Agitator Speed:	250 rpm
Pre Purge Time:	0 s
Injector:	Selected from the list of injectors (such as Injector1 or Injector2).
Injection Flow Rate:	25 mL/min
Post Purge Time:	60 s
Analysis Time:	Sum of GC program time, cooling time, and equilibration time, plus an extra margin.

Setup Group

Gas Chromatograph:	Selected from the list of GC units (such as GC1).
Sync Before Incubation End Time:	0 min
Agitator:	Selected from the list of agitators.
Do Agitation:	True
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:	Flase

Advanced Group

Enable Pre Filling:	True
Filling Strokes Count:	5
Filling Strokes Volume:	1.2 mL
Filling Strokes Aspirate Flow Rate:	6 mL/min
Delay After Filling Strokes:	30 s
Sample Aspirate Flow Rate:	6 mL/min
Sample Post Aspirate Delay:	0 s

Sample Vial Penetration Speed:	25 mm/s
Injector Penetration Speed:	25 mm/s
Pre Injection Dwell Time:	3 s
Post Injection Dwell Time:	10 s
Agitator On Time:	5 s
Agitator Off Time:	2 s

NOTE

Depending on the version of the AOC-6000 control software, the “Headspace Injection” method has different items in the [Advanced Group]. It is recommended that the default values for the parameters of the [Advanced Group] are used as they are. The parameter examples are shown for AOC-6000 control software version 2.0 or later.

NOTE

Set the injection volume to 500 µL for batch analysis.

注記

Set the split ratio to 5 or higher. The splitless mode or low split ratios can cause poor peak shape due to pressure fluctuations at the injection port.

- **ITEX Injection**

  Analysis Group

ITEX Tool:	Selected from the list of syringe tools for ITEX.
Trap Pre Cleaning Temperature:	250 °C
Trap Pre Cleaning Time:	300 s
Incubation Temperature:	80 °C
Incubation Time:	5 min
Agitator Speed:	250 rpm
Syringe Temperature:	90 °C
Trap Extraction Temperature:	40 °C
Extraction Strokes:	50
Injector:	Selected from the list of injectors (such as Injector1 or Injector2).

Injector Aspirate Flow Rate:	10 µL/s
Desorb Temperature:	250 °C
Desorb Flow Rate:	100 µL/s
Trap Post Cleaning Temperature:	250 °C
Trap Post Cleaning Time:	0 s
Analysis Time:	Sum of GC program time, cooling time, and equilibration time, plus an extra margin.

Setup Group

Gas Chromatograph:	Selected from the list of GC units (such as GC1).
Sync Before Incubation:	0 min
Agitator:	Selected from the list of agitators.
Heat Agitator:	True
Sample Vial Depth:	15 mm
Water Removal:	Disable
Water Removal Position:	Not selected.
Injection Signal Mode:	PlungerUp
Injector Penetration Depth:	33 mm

Advanced Group

Extraction Volume:	1000 µL
Extraction Aspirate Flow Rate:	100 µL/s
Extraction Dispense Flow Rate:	100 µL/s
Sample Prefill Rate:	40 %
Water Removal Trap Temperature:	60 °C
Water Removal Purge Time:	0 s
Injector Penetration Speed:	50 mm/s
Post Inject Delay:	0 s

NOTE

Set the injection volume to 500 µL for batch analysis.

NOTE

Set the split ratio to 5 or higher. The splitless mode or low split ratios can cause poor peak shape due to pressure fluctuations at the injection port.

NOTE

Select the type of trap sorbent used based on the targeted components.

Appendix 5 Using the Database with TD-30 Series Autosampler

This chapter describes the operating procedure when using an TD-30 series sampler as a pretreatment system. To use a TD-30 series thermal desorption unit, create a method file for the TD-30 unit.

> **Hint**
>
> For TD-30 series operating instructions, refer to the TD-30 Series Instruction Manual.

Appendix 5.1 Instrument Configuration Settings

Configure instrument configuration settings as described in 2.2 GC Set-up in the TD-30 Series Instruction Manual.

Appendix 5.2 Analyzing n-Alkanes

To use a TD-30 series system to analyze n-alkanes for retention time adjustment, measure the n-alkanes by adding them to TD sample tubes.

> **NOTE**
>
> Pre-condition sample tubes before analyzing n-alkanes and samples. For the tube conditioning procedure, refer to 5.6 Conditioning in the TD-30 Series Instruction Manual.

1) Use a microsyringe to add 1 µL of the n-alkane standard mixture sample (50 µg/mL) to the sample tubes. Place the sample tubes in the TD-30 sample tray.

2) Create a TD-30 method file and batch file for measuring n-alkanes as described in 4.1 Setting the Analytical Conditions in the TD-30 Series Instruction Manual. Use Appendix 5.4 Examples of Method Parameter Settings as a reference for specifying TD-30 method parameter settings.

Hint

If the GCMSsolution TD Add-in is installed, a TD-30 batch file does not need to be created because the TD-30 unit is controlled by GCMSsolution batch processing.

3) Analyze the n-alkane sample as described in 5.1. Starting Pretreatment in the TD-30 Series Instruction Manual.

Hint

If a TD-30 series system is used for semi-quantitative analysis according to the procedure in Appendix 3 Calculating Semi-Quantitative Results for Registered Compounds, then also analyze the internal standard substance using the same procedure as used for the n-alkanes.

Appendix 5.3 Analyzing Samples

1) Use the n-alkane measurement data to adjust the retention times in the method file as described in 2.1.4 Adjusting Retention Times and Analyzing Samples. If using a database to create the method, follow the procedure in 2.3 Using a Smart Database to Create Method Files, starting from 2.3.3 Specifying the Smart Database.

2) Create a TD-30 method file and batch file for measuring samples as described in 4.1 Setting the Analytical Conditions in the TD-30 Series Instruction Manual. For TD-30 parameter settings, refer to Appendix 5.4 Examples of Method Parameter Settings.

3) Measure samples as described in 5.1 Starting Pretreatment in the TD-30 Series Instruction Manual.

Appendix 5.4 Examples of Method Parameter Settings

Use the following examples as a reference for specifying method parameter settings for respective TD-30 sample injection methods.

- **TD-30R**

Pre purge flow:	20 mL/min
Pre purge time:	0 min
Tube desorption temperature:	280 °C
Tube desorption flow rate:	60 mL/min
Tube desorption time:	5 min
Trap cooling temperature:	-20 °C
Trap desorption temperature:	250 °C
Trap desorption time:	5 min
Joint temperature:	250 °C
Valve temperature:	250 °C
Transfer line temperature:	250 °C
GC Cycle Time:	Sum of GC program time, cooling time, and equilibration time, plus an extra margin.

Internal STD

Variable volume:	–

Restore

Restore:	–
Dry Purge:	–

Stand-by Temp.

Tube:	40 °C
Trap:	50 °C

- **TD-30**

Tube desorption temperature:	280 °C
Tube desorption flow rate:	60 mL/min
Tube desorption time:	5 min
Trap cooling temperature:	-20 °C
Trap desorption temperature:	250 °C
Trap desorption time:	5 min
Joint temperature:	250 °C
Valve temperature:	250 °C
Transfer line temperature:	250 °C
GC Cycle Time:	Sum of GC program time, cooling time, and equilibration time, plus an extra margin.

Stand-by temperature

Tube:	40 °C
Trap:	50 °C

Appendix 6 Using the Database with HS-20 Series Autosampler

This chapter describes the operating procedure when using an HS-20 series sampler as a pretreatment system. If using an HS-20 series headspace sampler, specify HS-20 method parameters in the method file to be used.

> **Hint**
>
> For HS-20 series operating instructions, refer to the HS-20 Operation Guide and HS-20 Control Software Instruction Manual.

> **NOTE**
>
> Due to the operating principle involved in analysis using headspace methods, components that are difficult to vaporize tend to be difficult to detect.

> **NOTE**
>
> Semi-quantitative calculations described in Appendix 3 are not available when using the HS-20 series system.

Appendix 6.1 Instrument Configuration Settings

Configure instrument configuration settings as described in the HS-20Control Software Instruction Manual.

Appendix 6.2 Analyzing n-Alkanes

If using an HS-20 series system to analyze n-alkanes for retention time adjustment, add n-alkanes to an empty vial.

1) Add 5 µL of the n-alkane standard mixture sample (1000 µg/mL) to an HS-20 vial. Place the vial in the HS-20 sample tray.

2) Specify method parameter settings according to the procedure 2.2 Method Development in

the HS-20 Control Software Instruction Manual. Use Appendix 6.4 Examples of Method Parameter Settings as a reference for specifying HS-20 method parameter settings.

3) Measure the n-alkane sample as described in 2.3 Batch Processing in the HS-20 Control Software Instruction Manual.

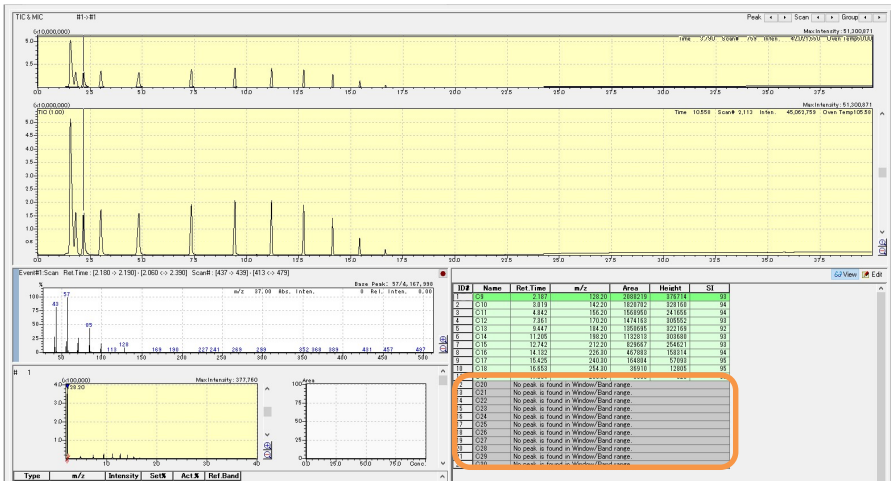
Appendix 6.3 Analyzing Samples

1) Use the n-alkane measurement data to adjust the retention times in the method file as described in 2.1.4 Adjusting Retention Times and Analyzing Samples. If using a database to create the method, follow the procedure in 2.3 Using a Smart Database to Create Method Files, starting from 2.3.3 Specifying the Smart Database.

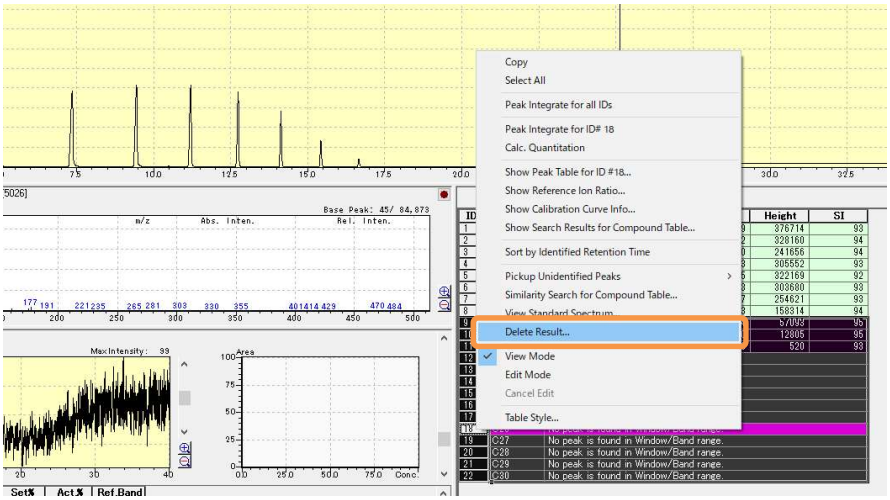
NOTE

If using an HS-20 system to measure n-alkanes, note that it cannot detect n-alkanes with all carbon numbers. If the vial is heated to 60 °C, then carbon numbers up to C18 can be detected by the trap mode and C16 by the loop mode. If undetected alkanes are misidentified, then delete those identification results according to the following procedure.

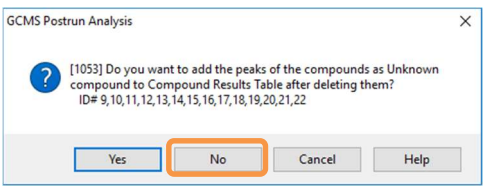
1. On the compound table [Results] tab page, select the alkanes that were not detected.



2. Right-click to select [Delete Result] on the right-click menu.



3. Click [No].



> **NOTE**
>
> AART-predicted retention times will tend to deviate more for compounds that have a retention index that is after detected alkanes.

2) Specify method parameter settings for the method adjusted or created in step 1), according to the procedure described in 2.2 Method Development in the HS-20 Control Software Instruction Manual. Use Appendix 6.4 Examples of Method Parameter Settings as a reference for specifying HS-20 method parameter settings.

3) Place the measurement samples in the HS-20 rack and start analysis using the method file from step 3).

Appendix 6.4 Examples of Method Parameter Settings

Use the following examples as a reference for specifying method parameter settings for respective HS-20 sample injection methods.

- **HS-20 (Loop Mode)**

Mode:	Loop
Oven Temp.:	60 °C
Sample Line Temp.:	100 °C
Transfer Line Temp.:	100 °C
Shaking Level:	Off
Multi Injection Count:	1
Pressurize Gas Pressure:	80 kPa
Equilibrating Time:	10 min
Pressurizing Time:	1 min
Pressure Equilib. Time:	0.1 min
Load Time:	1 min
Load Equilib. Time:	0.1 min
Injection Time:	3 min
Needle Flush Time:	5 min
GC Cycle Time:	Sum of GC program time, cooling time, and equilibration time, plus an extra margin.

- **HS-20 (Trap Mode)**

Mode:	Trap
Oven Temp.:	60 °C
Sample Line Temp.:	100 °C

Transfer Line Temp.:	100 °C
Trap Cooling Temp.:	-10 °C
Trap Desorb Temp.:	280 °C
Trap Equilib. Temp.:	25 °C
Shaking Level:	Off
Multi Injection Count:	5
Pressurize Gas Pressure:	80 kPa
Dry Purge Gas Pressure:	60 kPa
Equilibrating Time:	10 min
Pressurizing Time:	1 min
Pressure Equilib. Time:	0.1 min
Load Time:	1 min
Load Equilib. Time:	0.1 min
Dry Purge Time:	10 min
Injection Time:	3 min (n-Alkanes: 30 min)
Needle Flush Time:	5 min
GC Cycle Time:	Sum of GC program time, cooling time, and equilibration time, plus an extra margin.

Appendix 7 Using the Database with OPTIC-4 Series Inlet

This chapter describes the operating procedure when using an OPTIC-4 series inlet unit as a pretreatment system. To use an OPTIC-4 unit, create an OPTIC-4 method file.

Hint

For OPTIC-4 unit operating instructions, refer to the OPTIC-4 Instruction Manual.

Hint

For instructions on operating it in combination with an AOC-6000 series optional LINEX-2 system, refer to the separate LINEX Method Instructions for AOC-6000 Control Software Instruction Manual.

Appendix 7.1 Instrument Configuration Settings

Configure instrument configuration settings as described in 6. Basic Operations in the OPTIC-4 Instruction Manual.

Appendix 7.2 Analyzing n-Alkanes

If using an OPTIC-4 unit to analyze n-alkanes for retention time adjustment, use the liquid injection method.

1) Install an OPTIC-4 insert liner for liquid injection.

2) Create a TD-30 method file and batch file for measuring n-alkanes as described in 7. Creating and Executing Methods in the OPTIC-4 Instruction Manual. Use Appendix 7.4 Examples of Method Parameter Settings as a reference for specifying OPTIC-4 method parameter settings.

3) Use the OPTIC-4 method created in step 1) and the GCMS method created in step 2) to analyze the n-alkane standard mixture sample (50 µg/mL).

Hint

If an OPTIC-4 series system is used for semi-quantitative analysis according to the procedure in Appendix 3 Calculating Semi-Quantitative Results for Registered Compounds, then also analyze the internal standard substance using the same liquid injection method as used for the n-alkanes.

Appendix 7.3 Analyzing Samples

Revise the method parameter settings based on the sample injection method used.

1) Use the n-alkane measurement data to adjust the retention times in the method file as described in 2.1.4 Adjusting Retention Times and Analyzing Samples. If using a database to create the method, follow the procedure in 2.3 Using a Smart Database to Create Method Files, starting from 2.3.3 Specifying the Smart Database.

2) Replace the OPTIC-4 port insert liner based on the sample injection method used for sample analysis.

3) In the method adjusted or created in step 1), specify method parameter settings for the sample injection method used, according to the OPTIC-4 Instruction Manual. For information about OPTIC-4 method parameter settings, refer to Appendix 7.4 Examples of Method Parameter Settings.

4) Start analyzing the measurement samples using the method file indicated in step 3).

Appndix 7.4 Examples of Method Parameter Settings

Use the following parameter settings as a reference for specifying method parameter settings for respective OPTIC-4 sample injection methods. For more information about respective parameters, refer to 7.4 Method Parameters in the OPTIC-4 Instruction Manual.

- **Expert**

  General

Equilibration Time:	5 sec
End Time:	GC program duration

  Inlet Temperature

Initial Temperature:	40 °C
Delay Time:	0 sec
Ramp Rate 1:	50 °C/sec

Hold Temperature 1:	250 °C
Solvent Colling Effect:	No
Cooling Valve Mode:	No

Septum Purge Flow

Septum Purge Flow:	3 mL/min

Solvent Vending

Vent Mode:	Fixed Time
Vent Time:	0

If Using SH-I-5Sil MS or InertCap Pure-Wax Column

Column Flow / Inlet Pressure

Carrier Control Mode:	Pressure
Zero LINEX Head Pressure:	Yes
Initial Inlet Pressure 1:	83.5 kPa
Start Inlet Pressure 1:	83.5 kPa
End Inlet Pressure 1:	83.5 kPa

Split Flow

Direct Split Value Control:	No
Initial Split Flow:	5 mL/min
Split Flow 1:	5 mL/min

If Using SH-PolarWax Column

Column Flow / Inlet Pressure

Carrier Control Mode:	Flow
Zero LINEX Head Pressure:	Yes
Initial Column Flow 1:	1 mL/min
Start Column Flow:	1 mL/min
End Column Flow:	1 mL/min

Split Flow

Direct Split Value Control:	No
Initial Split Flow:	5 mL/min
Split Flow 1:	5 mL/min

Cryotrap (only if cryogenics is available)

Cryotap Low Temperature:	-150 °C
Low Temperature Hold Time:	300 °C
Cryotrap High Temperature:	60 °C/sec
Cryotrap Heat Ramp Rate:	250 °C

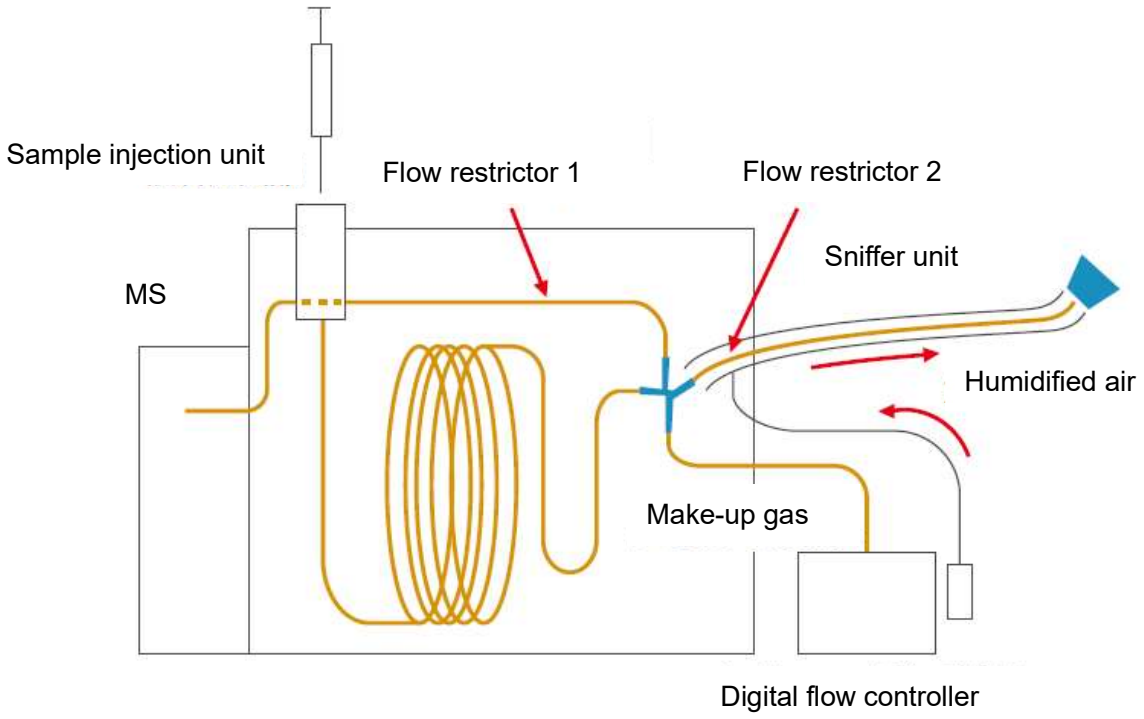
Appendix 8 Connecting an Odor Sniffer

> **NOTE**
>
> The retention index values included in the product were acquired using only a standalone GCMS system. Splitting flows between an odor sniffer unit and an MS unit can cause a large shift in the retention index values that varies depending on the compound.

Appendix 8.1 Flow Restrictor Settings for Connecting a Sniffer Unit

If connecting a sniffer unit to a 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 of components eluted from the column. The following split ratios are recommended for balancing the sensitivity of the MS unit with the flow rate of the sniffer unit.

	Internal Diameter (mm)	Length (m)	Connected to	Flow Rate Split Ratio (MS: Sniffer)
Flow Restrictor 1 (MS Flow)	0.15	1.7	MS	About 0.5:1
Flow Restrictor 2 (Sniffer Flow)	0.25	2.0	Sniffer unit	



Appendix 8.2 Methods for when a Sniffer Unit is Connected

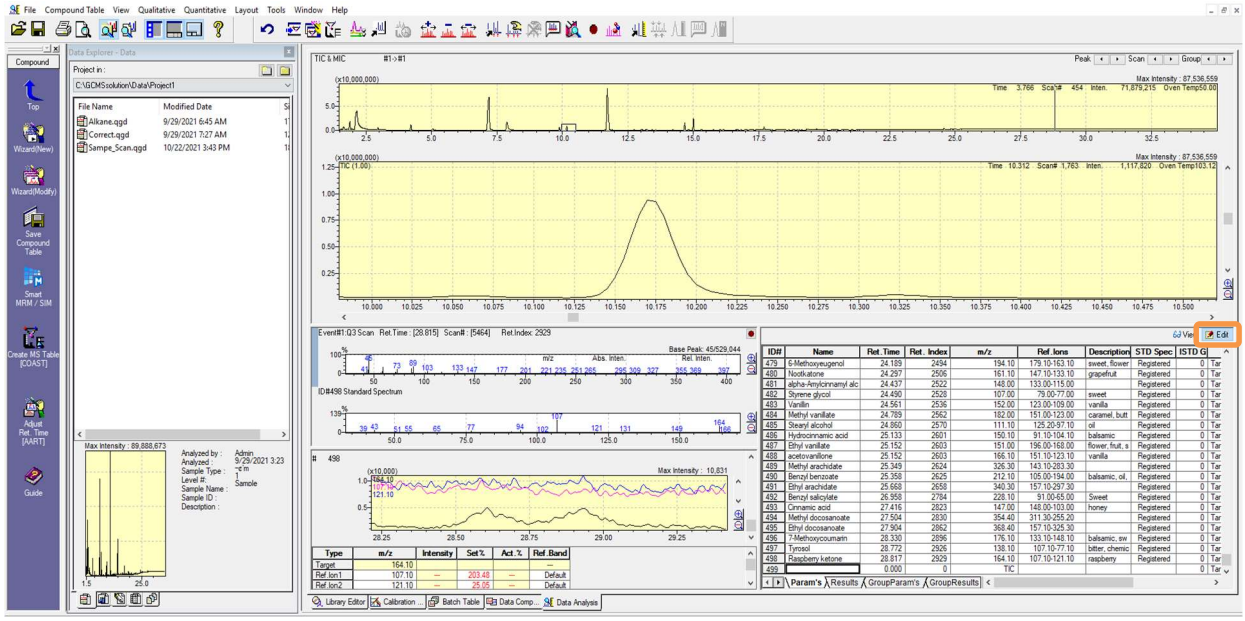
If a sniffer unit is connected, it applies pressure to the column outlet, which requires changing the injection port pressure setting in regular GC-MS methods. To analyze samples using a system with a sniffer unit connected, use a method file located in the folder below.
“C:\GCMSsolution\SmartDatabase\Aroma\<column name>\Sniff” folder

Column	File Name	Description
SH-I-5Sil MS	Aroma_QP/TQ_Sniff_SH-I-5SilMS_Scan.qgm	Method file for analyzing aromas/odors (Scan)
	Aroma_QP/TQ_Sniff_ SH-I-5SilMS_AART.qgm	Method file for retention time correction
	Aroma_QP/TQ_Sniff_SH-I-5SilMS_Correct.qgm	Method file for sensitivity correction analysis
	Aroma_QP/TQ_Sniff_SH-I-5SilMS_Template.qgm	Template for creating SIM method file from Smart Database
SH-PolarWax	Aroma_QP/TQ_Sniff_SH-PolarWax _Scan.qgm	Method file for analyzing aromas/odors (Scan)
	Aroma_QP/TQ_Sniff_SH-PolarWax_AART.qgm	Method file for retention time correction
	Aroma_QP/TQ_Sniff_SH-PolarWax_Correct.qgm	Method file for sensitivity correction analysis
	Aroma_QP/TQ_Sniff_SH-PolarWax_Template.qgm	Template for creating SIM method file from Smart Database
InertCap PureWAX	Aroma_QP/TQ_Sniff_IC-WAX_Scan.qgm	Method file for analyzing aromas/odors (Scan)
	Aroma_QP/TQ_Sniff_IC-WAX_AART.qgm	Method file for retention time correction
	Aroma_QP/TQ_Sniff_IC-WAX_Correct.qgm	Method file for sensitivity correction analysis
	Aroma_QP/TQ_Sniff_IC-WAX_Template.qgm	Template for creating SIM method file from Smart Database

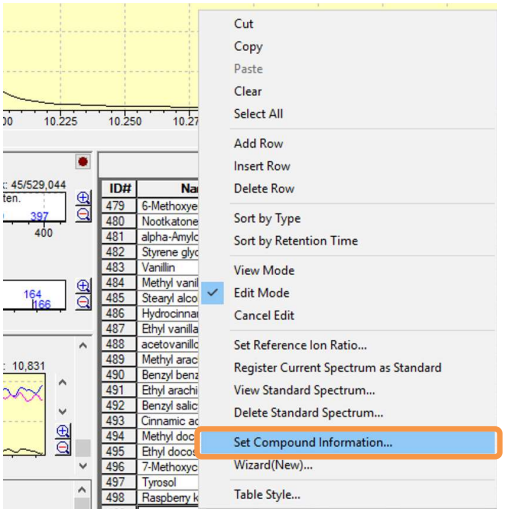
Appendix 9 Adding Target Compounds

Appendix 9.1 How to Add a Compound to a Method File (Scan)

1) Use a method file with adjusted retention times to measure a sample that contains the additional compound to be registered.
2) In the [GCMS Postrun Analysis] program, open the data file.
3) Click the compound table [Edit] button.

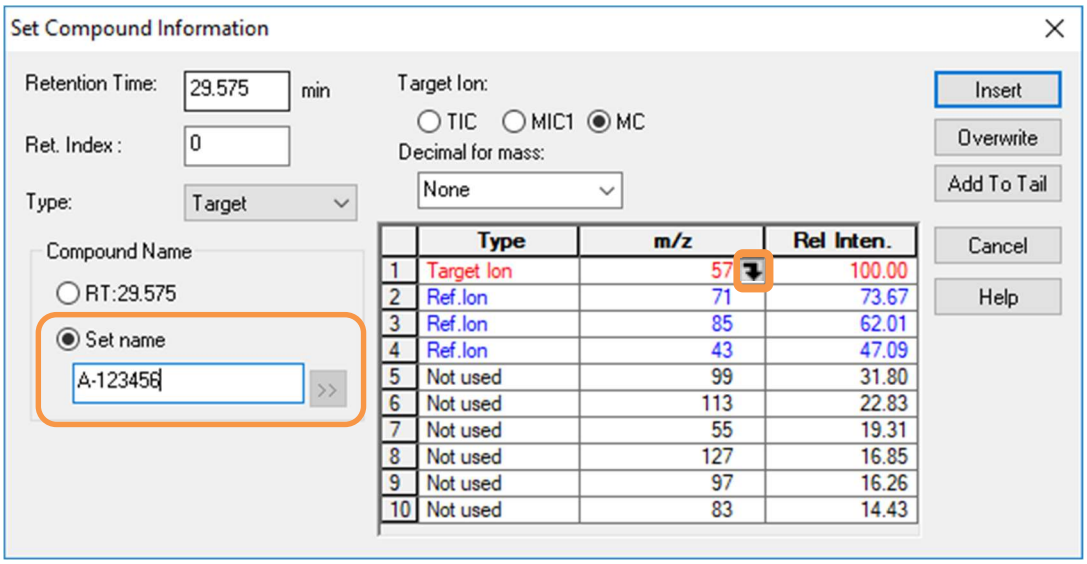


4) Left-click to select the last row of the compound table, where nothing is registered.
5) Select the peaks for the compound to register and, if necessary, subtract the background.
6) Left-click to select the last row of the compound table, where nothing is registered. Then right-click to click [Set Compound Information] on the right-click menu.

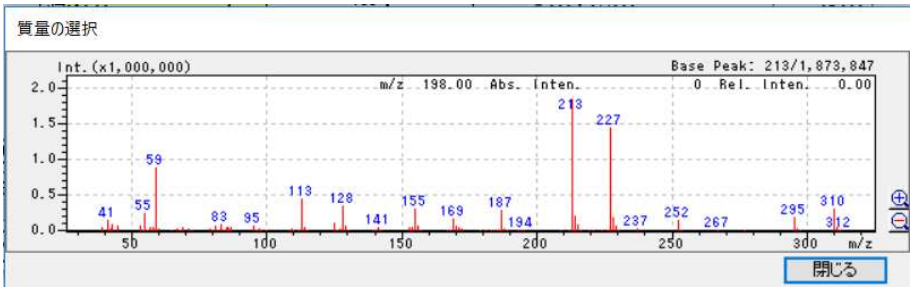


7) Select [Set name] and register the compound name.

Register the target and reference ion values. Generally, the ions with the highest relative intensity are registered automatically. To change a registered ion, click the [m/z] setting and then click the arrow button. That registers the mass spectrum.



8) In the mass spectrum, double-click on the peaks to register the corresponding ions to be specified.



9) Click [Overwrite] or [Add to Tail] to register the compound.

10) Save the method file with a new name.

11) If necessary, overwrite the data file.

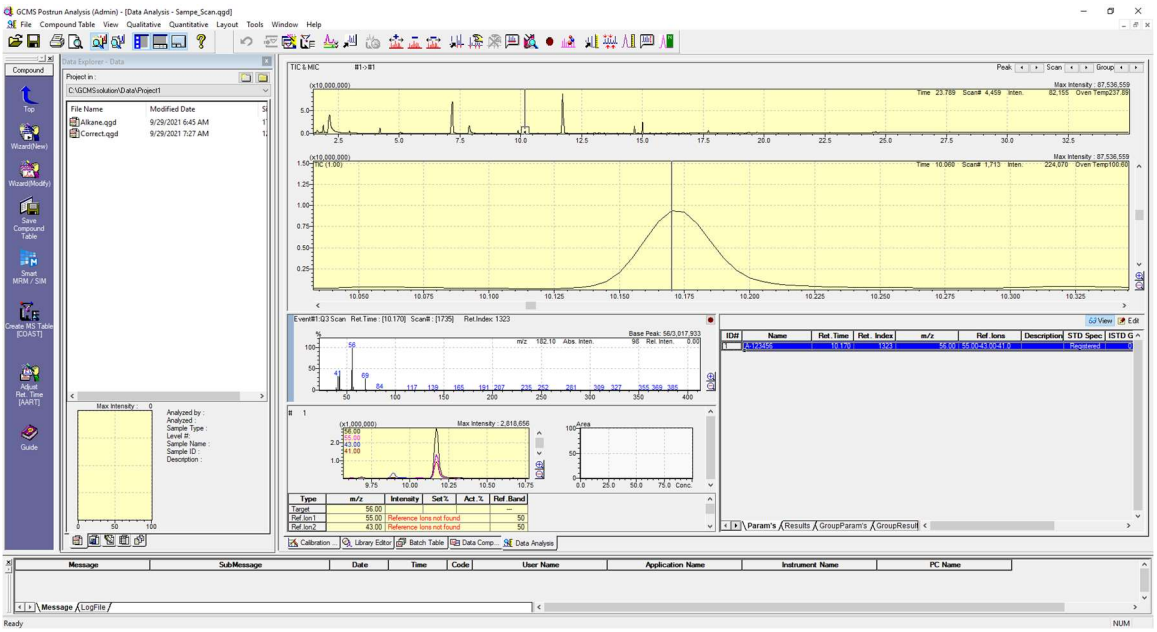
NOTE

Compound table information is saved in the method file. If it was not saved, open the data file again and save the method file.

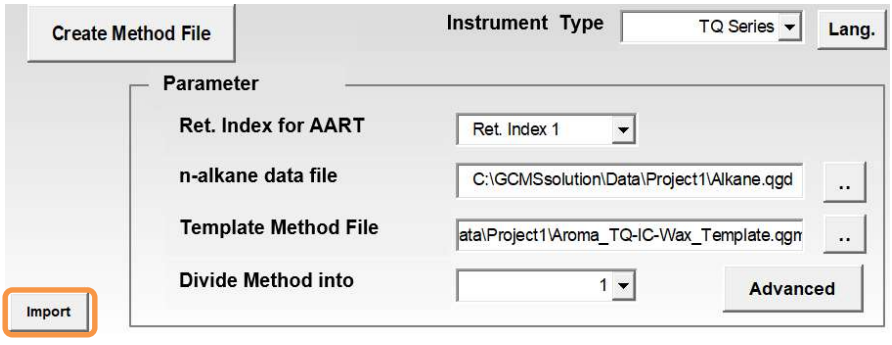
Appendix 9.2 How to Add a Compound to a Database File (Scan/SIM)

Compound information registered in method files is registered in a database file in the product. That database file can be used to create method files. Therefore, newly registered compounds can be managed more conveniently by registering them in a database file.

1) Register the compounds using the same procedure as described in 1. How to Add a Compound to a Method File.
2) Open the data file and click [Quantitative Parameters] on the [Quantitative] toolbar.
3) Click the [Quantitative] tab and change the [Quantitative Method] setting to [External Standard].
4) Click the compound table [Edit] button. Then delete all compounds except the compound to be registered.



5) Save the method file with a different name.
6) Open the database file.
7) Click [Import] to open the method file saved in step 5). The compound is registered automatically.



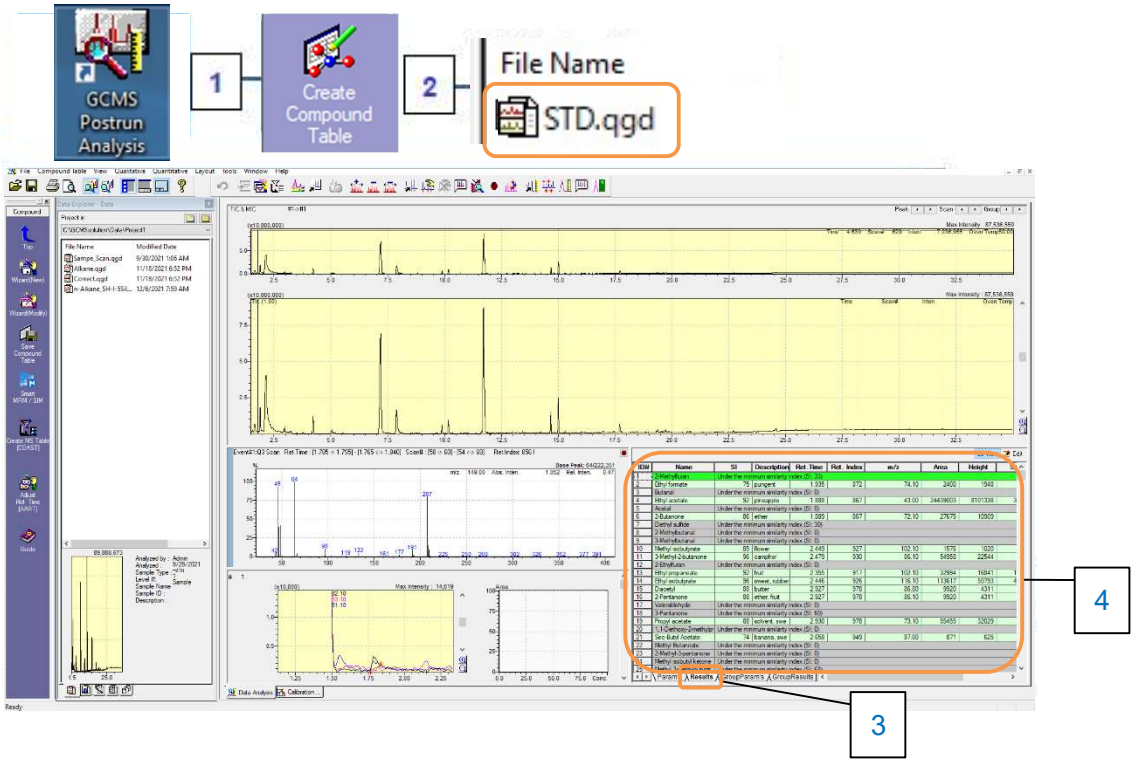
Appendix 9.3 How to Add a Compound to a Database File (MRM)

Register the information by following the series of steps described in 3. Registering Additional New Compound Information in a Smart Database in the GCMS Operation Guide – Method Development. After registering the information, complete the process by saving the database with a different name.

Appendix 10 Retention Time Adjustment Using a Standard Sample

1. Use the method file with adjusted retention times to measure the standard sample.

2. Identify target compounds.

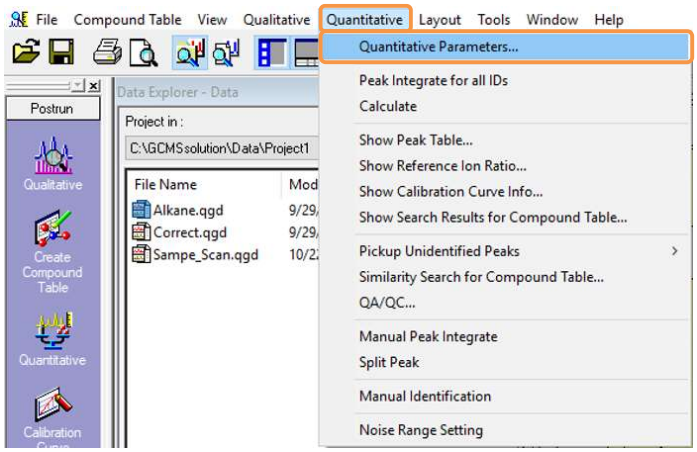


1) Start the [GCMS Postrun Analysis] program and then click the [Create Compound Table] icon on the [Postrun] assistant bar.
2) Open Data Explorer and click on the measurement data for the standard sample.
3) Click the [Results] tab below the compound table on the left.
4) Identify each component by checking the mass chromatogram (MC) in the quantitation view. If the components are not identified correctly, use manual peak integration.

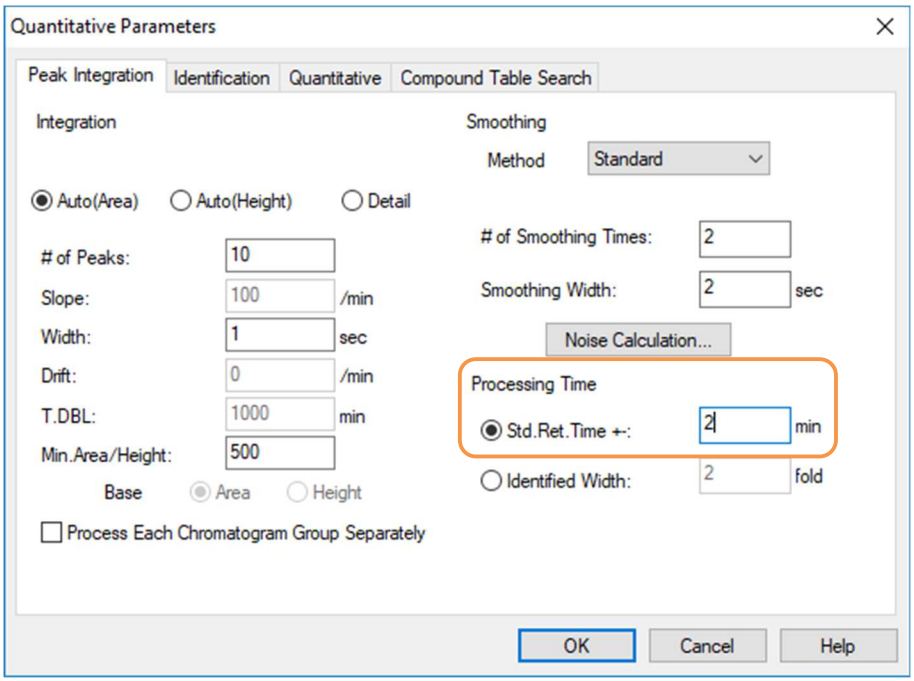
Hint

If the target compound peaks extend outside the quantitation view window, use the following procedure to specify a wider window for all compounds.

1) On the toolbar, click [Quantitative]-[Quantitative Parameters].

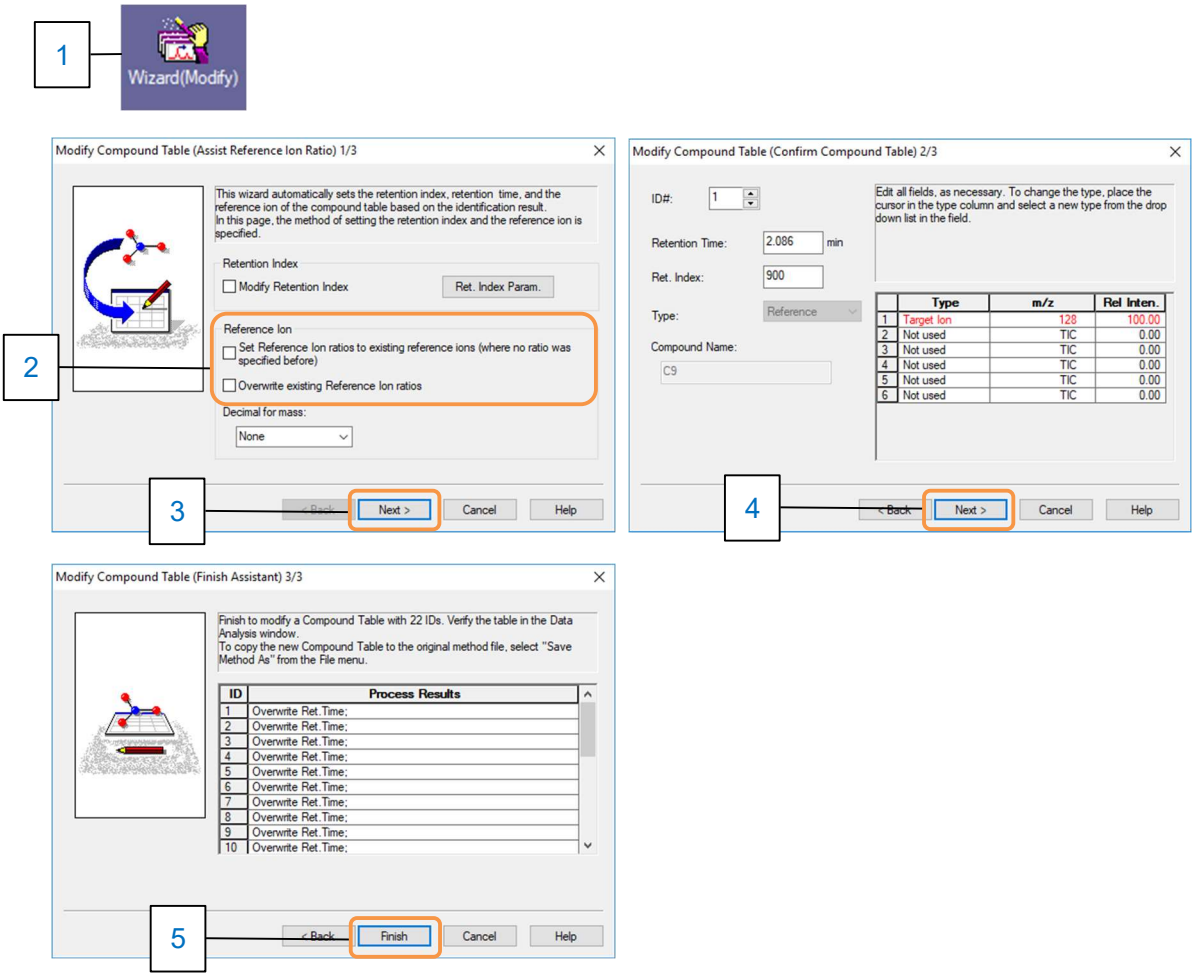


2) Set the [Processing Time]-[Std. Ret. Time+/-:] setting to 2 min or other suitable value and then click [OK].



3) That changes the window width for all compounds in the compound table based on the specified time range.

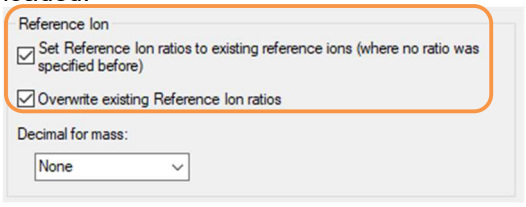
3. Use the [Wizard (Modify)] to apply the retention times from the identification results so they are reflected in the compound table.



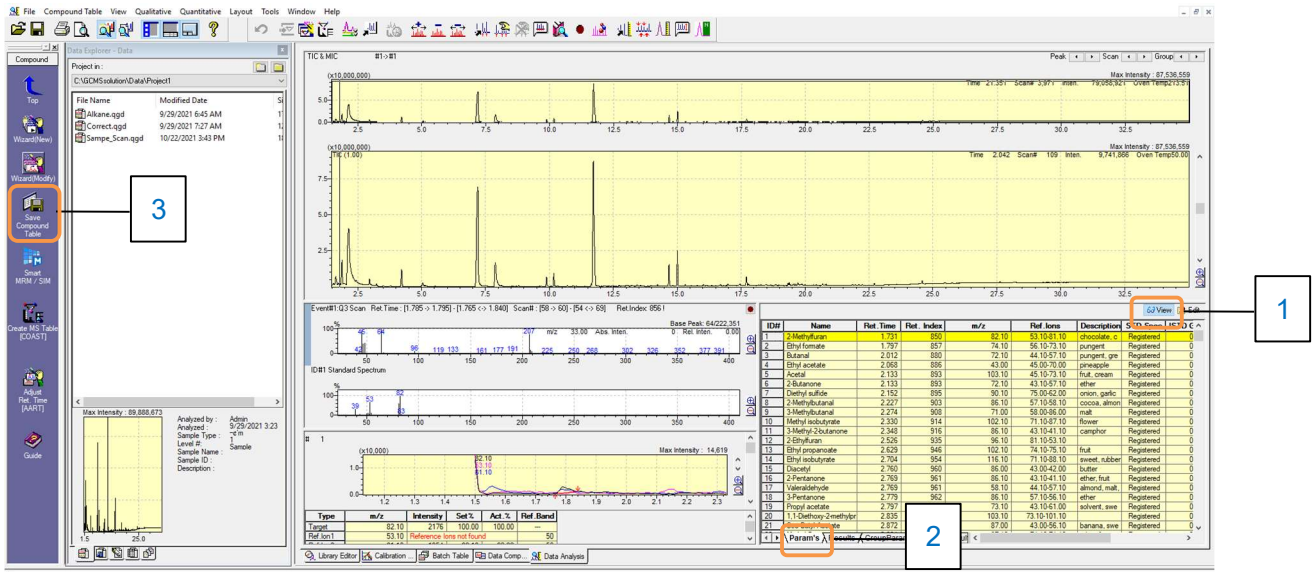
1) Click the [Wizard (Modify)] icon on the [Compound] assistant bar.
2) Clear the indicated checkboxes.
3) Click [Next].
4) Click [Next].
5) Click [Finish].

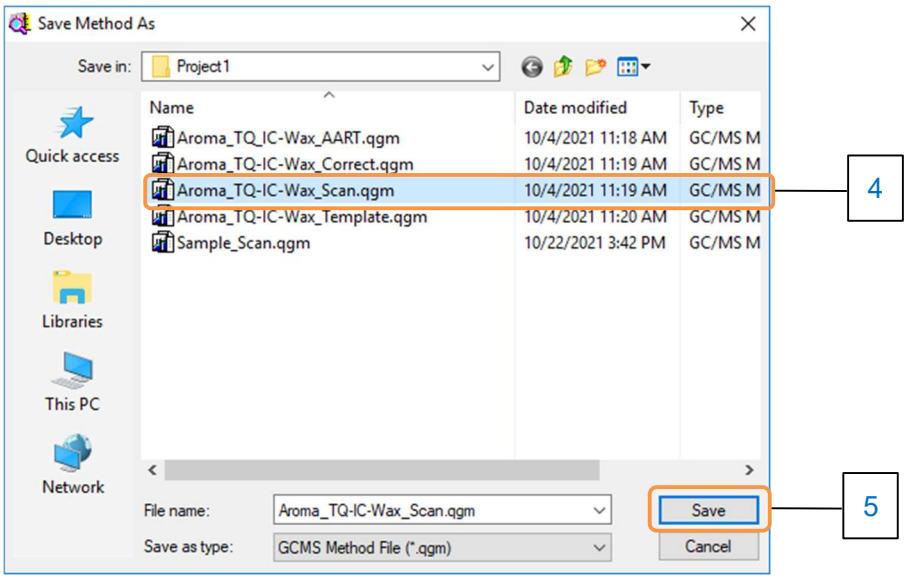
NOTE

About the [Modify Compound Table (Assist Reference Ion Ratio)] Window (1 of 3)
Normally, these checkboxes are cleared in step 2. Select these checkboxes if the reference ion ratios specified in the compound table need to be reset based on the data currently loaded.



4. Saving the Compound Table





1) Click the [View] icon located on the right side above the compound table.
2) Click the [Edit] tab on the left side below the compound table.
   That allows checking that the retention times are adjusted to the retention times in identification results.
3) Click the [Save Compound Table] icon on the [Compound] assistant bar.
4) Select the method file used for analysis.
5) Click [Save].

Appendix 11 Outputting Files for Statistical Data Analysis Software

To use statistical data analysis software for multivariate analysis or other statistical analysis, the area and area ratio values for respective peaks in multiple sets of data must be output in list form. Such data can be output as a CSV file using the following procedure in LabSolutions Insight.

1. Load the data to output.

1) Click [File]-[Open] 

on the menu bar to open the method file to be used for data analysis.

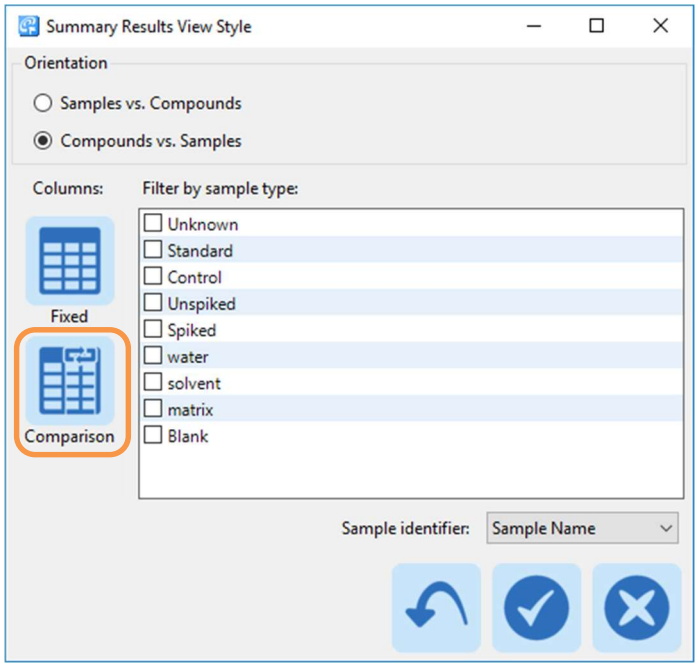
2) Click [File]-[Import] 

on the menu bar to load the data file to be output.

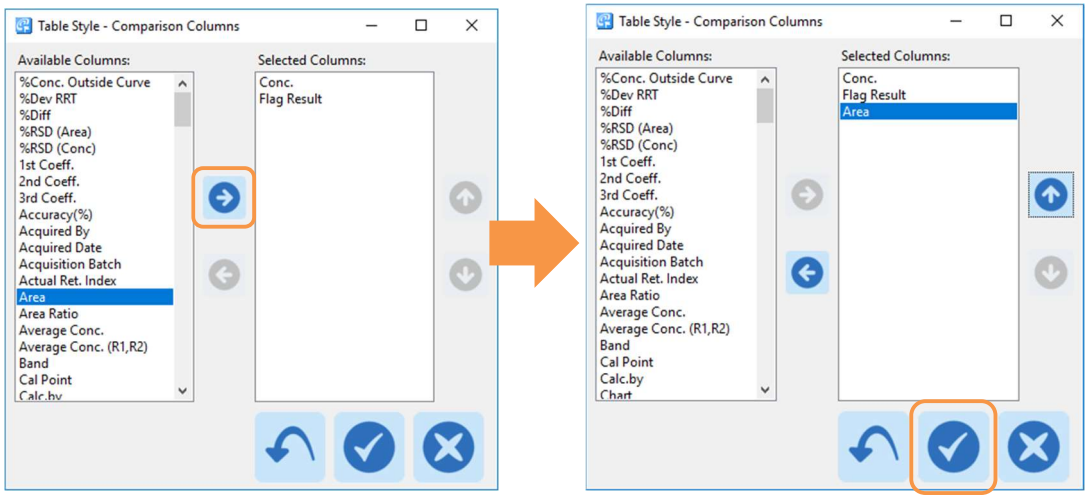
3) Check the data analysis results.

4) On the [View] menu, specify the [Summary] view. Right-click the caption in the summary pane and select [Properties].

5) In the [Summary Results View Style] window, click the [Comparison] icon.



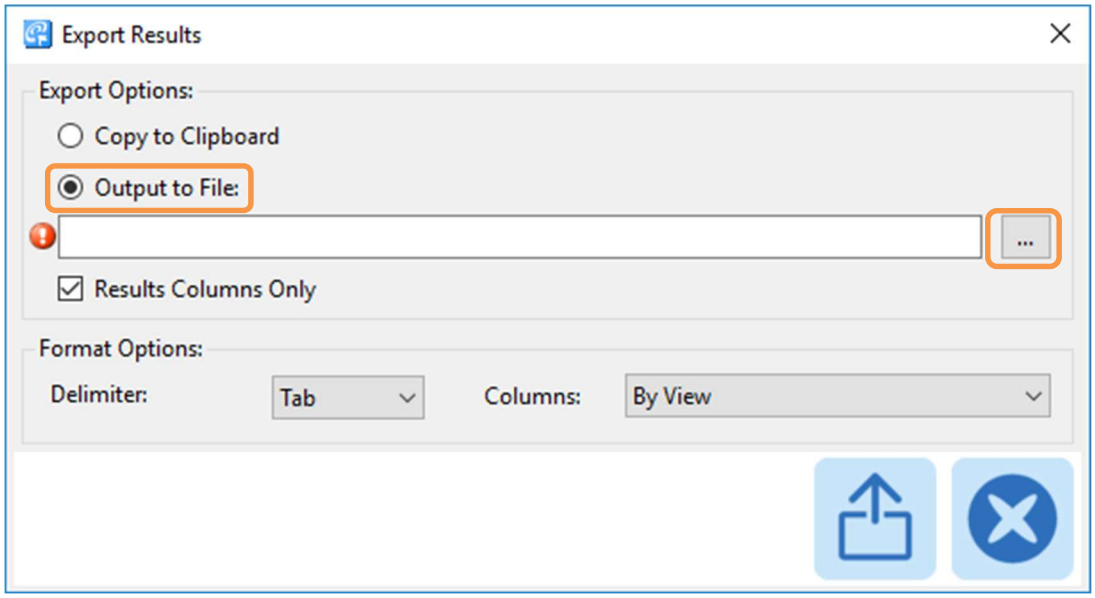
6) Use table style settings to specify the data to be output (Area, etc.) in the [Selected Columns] field. Then click the  (OK) icon.



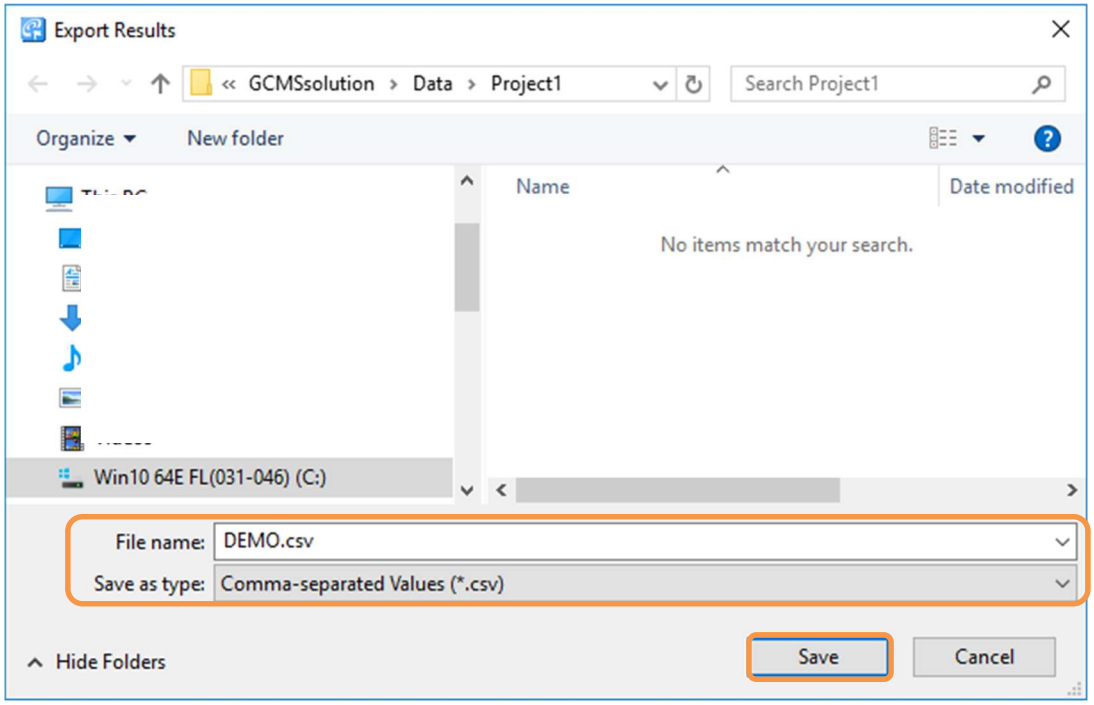
2. Output an Export File.

1) Click [File]-[Export] on the menu bar and then select [Results to File].

2) Select the [Output to File] checkbox and then click the […] (browse) button.


Export Results
Export Options:
Copy to Clipboard
Output to File:
...
Results Columns Only
Format Options:
Delimiter:
Tab
Columns:
By View

3) Specify the file name and click [Save as type]-[Comma-separated Values (*.csv)].



4) Click [Save]. That saves the data as a CSV file.

Appendix 12 Installing the Database

Follow the steps below to install the database.

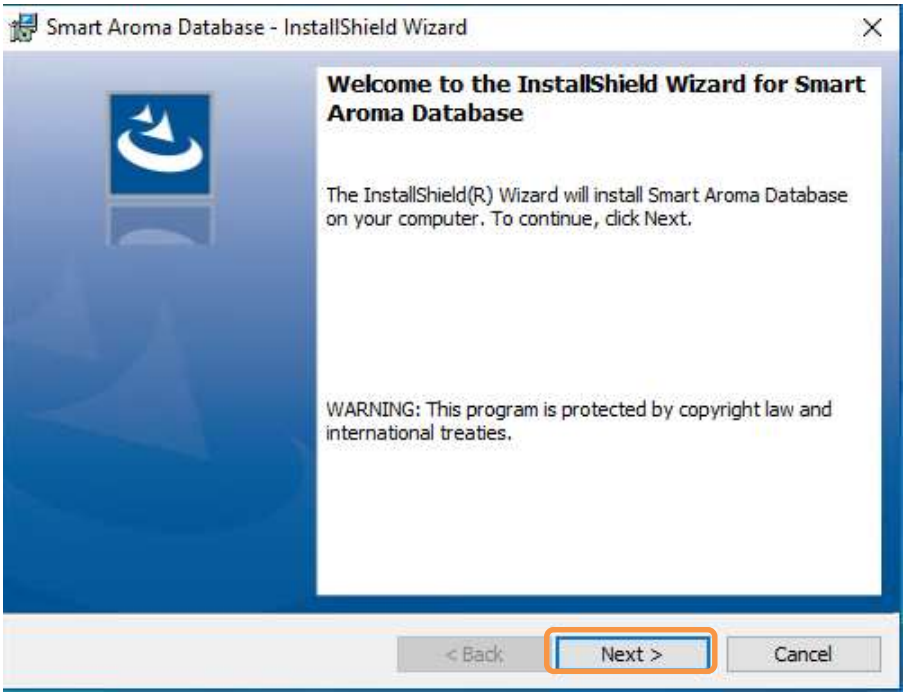
The explanations given here assume Windows 10 is used.

> **NOTE**
>
> GCMSsolution must already be installed before the database can be installed.

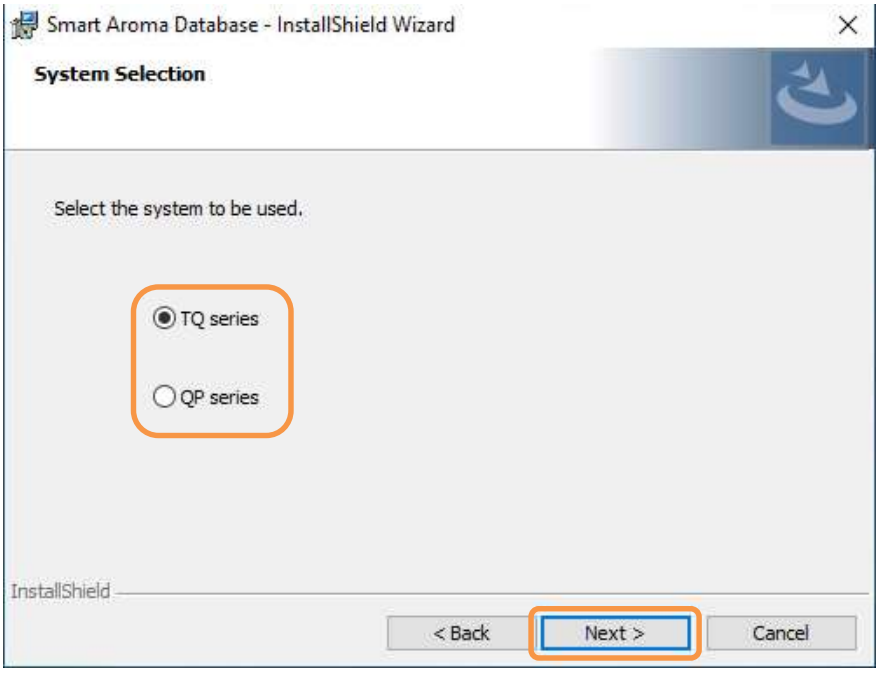
1) Insert the Smart Aroma Database installation disk into the CD-ROM drive. The installer program launches automatically.

> **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.

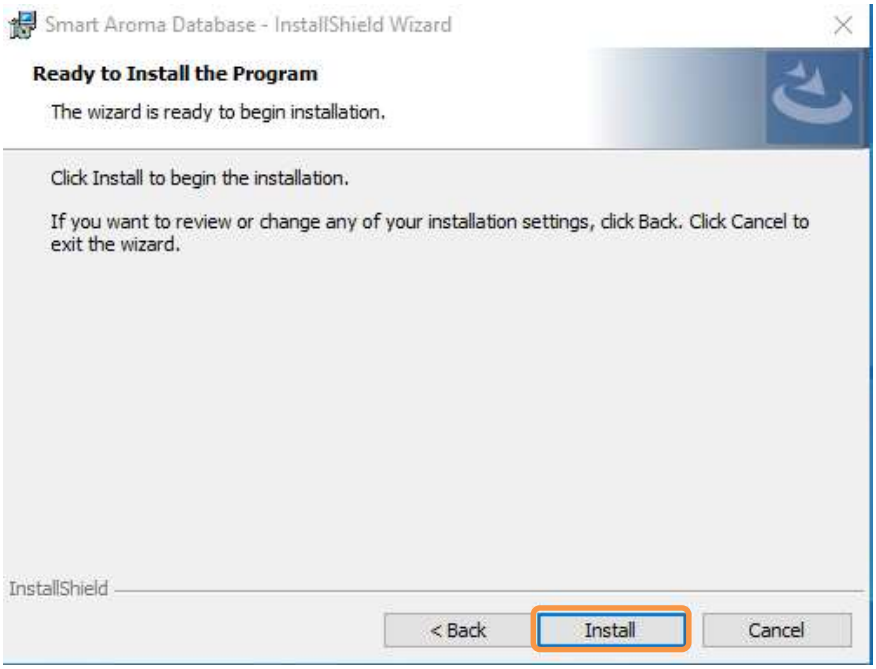
2) Click [Next>].



3) Select the MS model to be used. Click [Next>].



4) In the [Ready to Install the Program] window, click [Install] to start installing the various database files in the specified location.



Method files and database files are normally installed in the "C:\GCMSsolution\SmartDatabase" folder.

5) When installation is finished, the [InstallShield Wizard Completed] window is displayed. Click [Finish] to complete the process.