225-18120D
May 2008

Gas Chromatograph
Mass Spectrometer
GCMS-QP2010

System User's Guide
(for GCMSsolution Ver. 2.5)

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

SHIMADZU CORPORATION
KYOTO JAPAN
ANALYTICAL & MEASURING INSTRUMENTS DIVISION

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- Information in this publication is subject to change without notice and does not represent a guarantee on the part of the vendor.
- If the user or usage location changes, ensure that this Instruction Manual is always kept together with the product.
- To ensure safe operation, contact your Shimadzu representative if product installation, adjustment, or re-installation (after the product is moved) is required.
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Warranty Information

Thank you for purchasing the GCMS-QP2010.
Shimadzu provides the following warranty for this instrument.

1. Period

One year from the date of first use (limited to domestic use) or 6 months after repair or overhaul of the rotary pump or turbo molecular pump.

2. Terms

The manufacturer will provide free replacement parts for, or repair free of charge, any instrument that fails during the warranty period, if the cause can be attributed to a defect in manufacturing.

3. Items Not Covered by the Warranty

The warranty does not cover malfunctions that result from:

a) misuse;
b) repairs or modifications made by any company other than an authorized Shimadzu representative;
c) external factors;
d) operation under severe conditions, such as: environments with high temperature, high humidity, corrosive gas, vibration, etc.;
e) fire, earthquake or other forces of nature;
f) moving or transporting the instrument after its initial installation.
g) The warranty does not cover replacement of consumable items or parts that can be regarded as consumable.

Note

Floppy disks, CD-ROMs and other recording media are considered consumable parts.

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Replacement Parts Availability

Replacement parts for this instrument will be available for a period of seven (7) years after the discontinuation of the product. Thereafter, such parts may cease to be available.
Please note that Shimadzu does not manufacture some parts of the instrument. For such parts, we will estimate the volume of part use within the aforementioned period after receiving notification of product discontinuation, and we will stock the part. However, due to incorrect estimates or circumstances under the control of the parts manufacturer, parts not manufactured by Shimadzu may not be available during the 7-year period after discontinuation of the product.

Precautions for Safe Operation

The gas chromatograph mass spectrometer is an analytical instrument used for qualitative and quantitative analyses.

Please note the following points to facilitate safe operation of this instrument.

1. Use this instrument only for the specified types of analyses.
2. Follow the procedures as written in this manual.
3. Observe all warnings and precautions.
4. Do not disassemble or modify the instrument without the express approval of an authorized Shimadzu representative.
5. For service or repair, contact your Shimadzu representative.

Note

Warnings, precautions and other items of interest are indicated by the following conventions:

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.

Note

In this manual and on the instrument, the following symbols are used as indicated below.

Indicates danger from high voltage.

Indicates danger from high temperature components.

Refer to the user manual for details about handling or operation.

Warning

Internal repair of the instrument is dangerous. Contact specially trained Shimadzu representatives for service or repair.

Warning

Do not disassemble or modify the instrument without authorization, as this will compromise safety.

Warning

Read this user manual to learn how to safely maintain and operate the instrument, and follow all procedures as described. Operating the instrument other than as described is potentially hazardous.

Installation Site Precautions

Warning

The solvents used with the gas chromatograph mass spectrometer are flammable and toxic. Install the instrument in a well-ventilated room. Otherwise, solvent vapors could cause poisoning, or ignite and cause a fire. Do not use this instrument in an area that contains combustible gases, as the instrument may ignite them and cause a fire.

Warning

Do not place flammable materials near the column oven exhaust at the back of the instrument, as they could ignite and cause a fire.

Warning

The lab table or other surface on which this instrument is installed should be level, stable, and sufficiently strong to support the instrument's weight. Otherwise, the unit could tip over or fall off the surface.

Warning

Avoid installing the instrument where there is corrosive gas or excessive dust. The instrument performance could be affected and its service life shortened.

Installation Precautions

Warning

A Shimadzu engineer must perform instrument installation and configuration. To prevent potential injuries, contact a Shimadzu representative if the instrument must be moved after installation.

Power Supply

Power Requirements

For 100 V - 115 V Type

AC Single Phase, Frequency 50 - 60 Hz

GC:	1800 VA
MS:	1000 VA
PC (including CRT):	See PC manual.
Printer:	See printer manual.

For 220 V - 240 V Type

AC Single Phase, Frequency 50 - 60 Hz

GC:	2600 VA
MS:	1000 VA
PC (including CRT):	See PC manual.
Printer:	See printer manual.

Warning

Use a power supply with a circuit breaker that is dedicated to this instrument. Avoid sharing a power supply with other instruments. Do not use power cords other than those supplied with the instrument, as this may cause fire or electric shock.

- Please note that additional options will increase the required current capacity.

Warning

Ground the instrument. Grounding is necessary to prevent electric shock in the event of an accident or electrical short. Ground should be 100 Ω or less.

Warning

Do not place heavy objects on the power cable, and avoid placing hot items near the cable. Do not excessively bend or pull on the power cable. It could be damaged, resulting in fire or electric shock. If the cable becomes damaged, contact your Shimadzu representative.

Ambient Environment

Temperature: 18 - 28 °C and should be held constant (Specification warranty range); 15 - 35 °C (Running warranty range)
Humidity: 40 - 70 % (avoid condensation)

Note

Choose an installation site with minimal detrimental factors such as dust, vibration, ambient noise, corrosive gas, and magnetic fields. To maintain good performance, note the following directions.

1. Minimize temperature fluctuation during use.
2. Avoid exposure to hot or cold air.
3. Avoid exposure to direct sunlight.

Although this instrument can operate within the running warranty range, extensive operation beyond the specification warranty range may result in problems such as shortening the service life of the instrument.

Gases

Carrier gas: Helium
Supply pressure: 300 - 980 kPa
Purity: 99.995 % or greater

- The pressure and flow rate setting range on the GC varies according to the supply pressure. Normally a supply pressure of 700 - 800 kPa is necessary.
- Some optional accessories may require a gas other than the one specified above. Consult the user manual accompanying the accessory for more information.
- Some applications, such as agrochemical analysis, may require helium of a higher purity (99.999 - 99.9999 %).

Note

Helium of at least the purity indicated above must be supplied as the carrier gas through the supplied tubing. If a carrier gas of lesser purity is used, performance may be compromised. Refer to Section 2.3 "Gas Requirements", page 13 for more information.

High-Pressure Gas Precautions

Warning

A high-pressure gas cylinder will be used to supply the carrier gas. When handling the gas cylinders, observe the following suggestions.

1. Keep gas cylinders in a well-ventilated area outside of the instrument installation site. Avoid exposure to direct sunlight. Use lines to transport the gas from the cylinders to the instrument. For flammable gases, this precaution is required by law.
2. Ensure that gas cylinder temperature never exceeds 40 °C. No open flame permitted within 2 m of the cylinder.
3. Choose an instrument installation site with sufficient ventilation, and include checking for gas leaks with leak detection solution in your daily inspection procedure. Do not smoke or use open flames within 5 m of the instrument when using highly combustible gases, such as acetylene and hydrogen, or potentially combustible gases, such as oxygen and nitrous oxide. Install and maintain effective fire extinguishers.
4. Secure cylinders with clamps or by some other method to prevent them from falling over.
5. When finished using the gas, immediately close the main cylinder valve.
6. Verify that the pressure gauges are functional at least once every three months.
7. Warning signs (adhesive aluminum plates) are available to indicate hydrogen gas use. Ask your Shimadzu representative for more details. Signs are supplied free of charge to sites in which they are mandatory.

Legal authorization is required to use cylinders with a capacity of 300 m^3 or greater.

Please refer to high-pressure gas control laws, liquid petroleum gas safety regulations, general high-pressure gas safety regulations, and fire safety laws for more information.

For Safety Use of Hydrogen Carrier Gas

Warning

Hydrogen gas, which is used as the carrier gas in gas chromatographs, is a dangerous gas that explodes easily. Read safety precautions in "GCMS-QP2010 Series Hydrogen Carrier Gas Safety" thoroughly before using the GCMS-QP2010, and use the product correctly. Be sure to observe the precautions described there. They are important for ensuring safety. Cautions related to hydrogen gas are also listed in the gas chromatograph user manual. Please read these as well.

Hydrogen Gas Properties

Hydrogen is a dangerous gas which can easily explode if handled improperly. When hydrogen is to be used as the carrier gas, be sure to understand the characteristics and proper handling of this gas.

- Colorless, odorless gas
- Wide combustion range, combustible within range of 4 vol% to 75 vol% when mixed with air.
- Ignites with extremely low energy.
- Easily accumulates near the ceiling, as it is lighter than air.
- Quickly mixes with air due to its high diffusion rate.
- Automatically ignites when rapidly expanding.
- The hydrogen flame is invisible, and easily carried by the wind.
- When mixed with a halogen gas such as chlorine, it explodes on direct exposure to sunlight.

Operation Precautions

Warning

Always wear safety glasses or goggles when handling solvents. If solvent gets into the eyes, blindness could result. Should solvent get into the eyes, immediately flush with large amounts of water and seek medical attention.

Warning

Safety glasses should be worn during sample injection to prevent the dangers associated with mechanical injury from the syringe or solvents splashing into the eyes. The injection port is typically under a pressure of a few hundred kPa to enable carrier gas to flow into the column. As a result, when a liquid sample is injected with a syringe, the plunger may be discharged from the syringe barrel, or the flow may be reversed and the sample ejected. Furthermore, when a large sample volumes are injected, the septum deteriorates more quickly and the sample may be ejected from the injection port.

Warning

Do not place solvents near PCs, printers or other instruments, as fire or instrument damage could result.

Warning

Do not use flammable sprays (hair sprays, insecticide sprays, etc.) near this instrument, as they could ignite and cause a fire.

Warning

To prevent injuries and instrument damage, do not disassemble or modify this instrument, or perform internal repairs.

Warning

If the power cable becomes dusty, remove the plug from the outlet and wipe away the dust with a dry cloth. Fire may result if dust is allowed to accumulate.

Precautions for Inspection, Maintenance, Adjustment, and Care of the Instrument

Note

To clean the surface of the instrument, use a soft cloth and water or neutral detergent. Wipe dry with a clean cloth.

Note

Periodic maintenance of the rotary pump and turbo molecular pump are recommended. Refer to respective manual for the interval.

Warning Labels

Caution

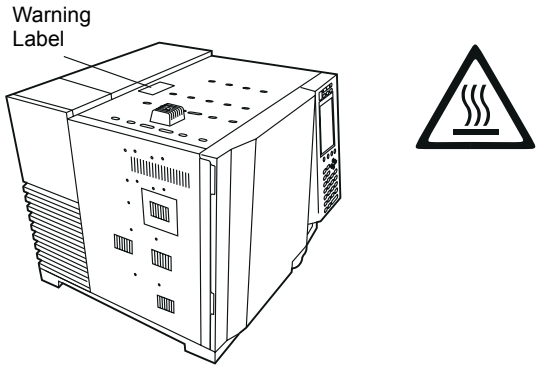
DO NOT BLOCK VENTS

Do not place anything on top of the instrument which might block vent holes and cause overheating.

Warning

DO NOT TOUCH

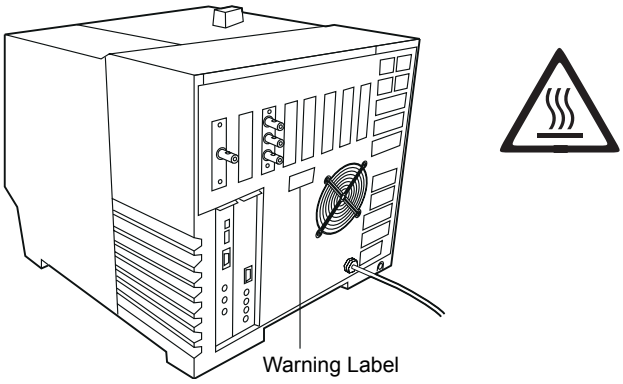
High Temperature injection ports, detectors and upper cover.



Warning

DO NOT TOUCH

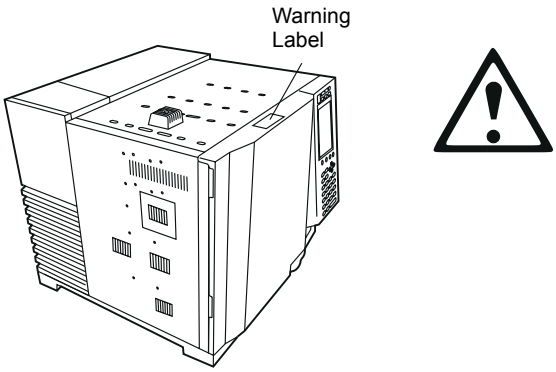
The rear panel may be hot and can cause burn.



Warning

PRECAUTIONS IN USING HYDROGEN GAS

Shut off hydrogen and cap unused column fittings to prevent accumulation of hydrogen in oven and possible explosion.

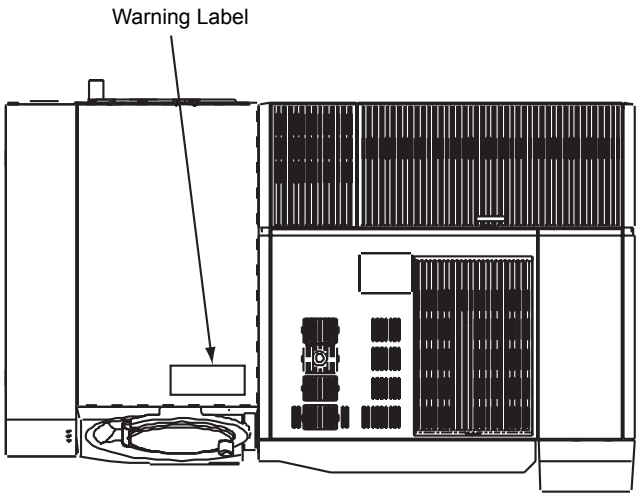


Warning

PRECAUTIONS IN USING HYDROGEN GAS

If hydrogen gas accumulates inside the vacuum chamber, it could potentially ignite.

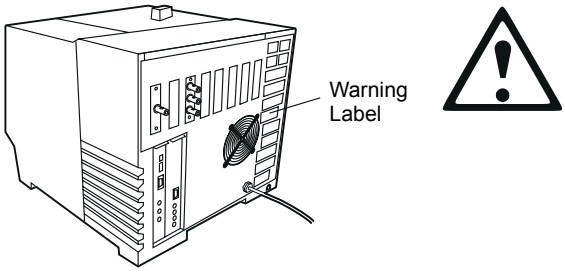
When hydrogen carrier gas is used, keep the knob on the front door completely loosened except when starting up the vacuum pump.



Warning

HOT AIR EXHAUST

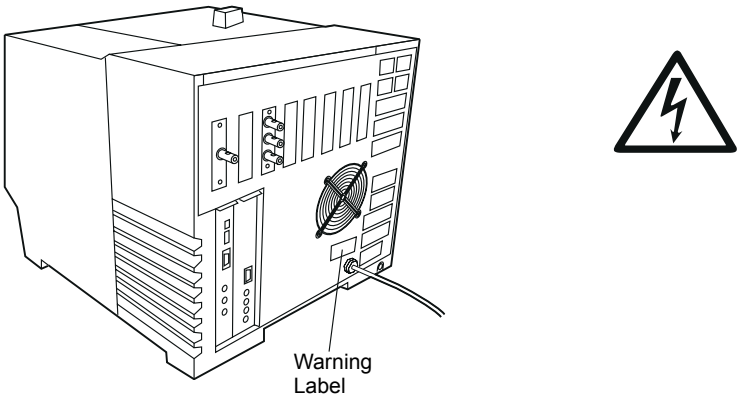
Keep temperature sensitive materials away from opening.



Warning

HIGH VOLTAGE

Disconnect power cable before removing cover. Refer servicing to qualified service personnel.



Warning

WARNINGS IN USING HYDROGEN GAS

When hydrogen gas is in use, care should be exercised in order to prevent accident.

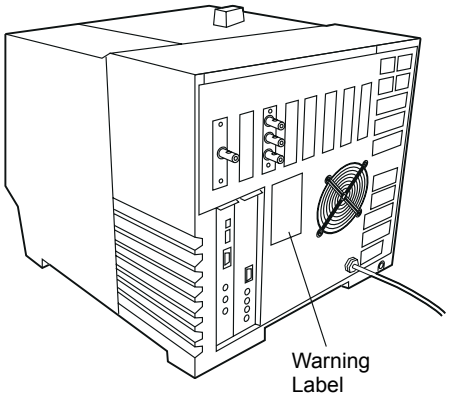
1. Connect gas lines correctly. Do not connect the hydrogen line to the air inlet, or hydrogen will leak excessively.
2. When the device is not in use, the main valve of the hydrogen gas cylinder must be closed. Also make sure there is no gas leakage from the main valve of the supply.
3. The flow line for hydrogen gas should be checked leakage whenever it is used.
4. To prevent buildup of explosive concentration in case the hydrogen gas leaks, the room in which the device is used should be ventilated.
5. When analyses are completed, close the main valve of the hydrogen gas container immediately before performing other procedures.

Caution

CAUTION IN GAS PLUMBING

The pressure regulators will be damaged when subjected to pressure beyond their specification.

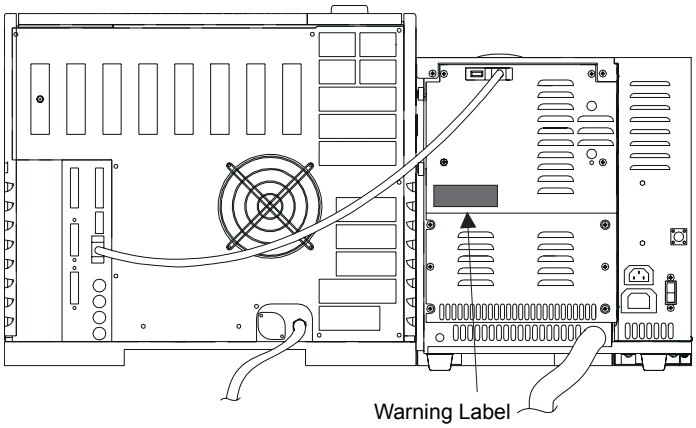
Do not exceed maximum source pressure of 800 kPa or the maximum specified pressure given in the instruction manual.



Warning

HIGH VOLTAGE

Do not open the cover. Danger! High voltage may cause electric shock.



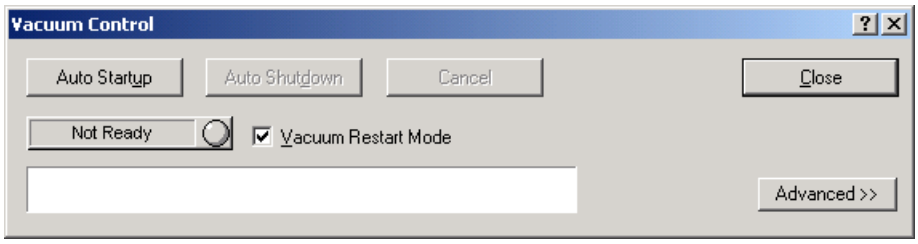
Handling Emergencies

The following measures should be taken in the event of an emergency such as a malfunction of the gas chromatograph mass spectrometer.
Take adequate precautions and contact your Shimadzu representative as necessary before resuming use of the instrument.
In the event of an emergency...

1. Turn off the gas chromatograph and mass spectrometer.
2. Turn off all accessories.
3. Close the valves in all carrier gas and air lines.
4. Turn off the power supply.
   - If the power cable is attached to a power board, turn off the power board.
   - If the power cable is plugged into an outlet, unplug the cable.

Measures during Power Outage

For power outages of 10 minutes or less, normally no particular procedure is required as long as the "Vacuum Restart Mode" checkbox is selected in the "Vacuum Control" window. The instrument will automatically restart when the power comes back on.



For long-time power outages, perform the following procedure.

Shutdown Operation for Long-Time Power Outages

1. Turn off the gas chromatograph and mass spectrometer.
2. Turn off all accessories.
3. Close the valves in all carrier gas and air lines.
4. Loosen the nut fastening the column on the interface to release the vacuum of the vacuum system.
5. Turn off the power supply.
   - If the power cable is attached to a power board, turn off the power board.
   - If the power cable is plugged into an outlet, unplug the cable.

For power outages while using hydrogen carrier gas, perform the following procedure.

Power Outage Operation While Using Hydrogen Carrier Gas

1. Immediately stop the supply of hydrogen gas.
2. Switch OFF power to the GC and the MS.
3. Open the windows and doors of the room where the instrument is installed to thoroughly ventilate.
4. Confirm that there is no ignition source in the room that could ignite the hydrogen gas.
5. Wait until the temperature of all GCMS-QP2010 components has fallen to the ambient temperature (approximately 1 hour).
6. Open the front door of the GC and loosen the nut of the interface to return the interior of the MS to atmospheric pressure.
7. Confirm that the knob on the front door of the MS is completely loose.
8. Wait until all hydrogen gas is completely expelled (approximately 30 minutes).
9. After confirming the items in "Precautions in Installation" and "Precautions in Operation and Running of the Instrument", use the standard startup procedure to start the GC and MS.

Action for Environment (WEEE)

To all users of Shimadzu equipment in the European Union:

WEEE Mark

Equipment marked with this symbol indicates that it was sold on or after 13th August 2005, which means it should not be disposed of with general household waste. Note that our equipment is for industrial/professional use only.
Contact Shimadzu service representative when the equipment has reached the end of its life. They will advise you regarding the equipment take-back.

With your co-operation we are aiming to reduce contamination from waste electronic and electrical equipment and preserve natural resource through and recycling.
Do not hesitate to ask Shimadzu service representative, if you require further information.

Voltage Fluctuation

To all users of Shimadzu equipment in the European Union:

This equipment can only be connected to a supply with the impedance 0.1 ohm or lower. If necessary, determine the impedance in consultation with the supply authority.

Note

If the actual system impedance Z_{act} exceeds 0.1 ohm at the point of interface point on the user's premises, the supply authority may impose restrictions to connection on the use of the equipment.

Note

If the actual system impedance Z_{act} has been declared to, or measured by the user, the user can use the information to assess the equipment's suitability without reference to the supply authority.

Introduction

Thank you for purchasing a GCMS-QP2010 instrument. Read this user manual carefully before using the instrument for optimal performance and to make full use of its capabilities.

This manual assumes that the user has a working knowledge of Windows. There are numerous references to functions and terminology specific to Windows; refer to a Windows user manual as necessary. Users who have never worked with Windows should read a Windows user manual before using this document.

Using this Manual

Read and understand this manual before operation. Keep this manual in a readily accessible location.
Window and dialog box names are italicized.
Menu names and commands, as well as Assistant Bar icon names are shown in bold.
A menu sequence is represented by the menu name (or item name) followed by a ">" followed by the item name (or sub-menu name) as in the following examples:

Main Menu Item > Sub-Menu Item > Selected Menu Item
Start > Settings > Control Panel
File > Print

When a menu sequence leads to a tabbed dialog box, the tab to select is shown in the same manner:

Method > Set Parameters > General tab

PDF versions of this guide and the operation guide, etc. should have been placed in the folder "GCMSsolution\Manual" on your computer. Please refer to them as necessary.

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CONTENTS

1 Overview

1.1 GCMS-QP2010

This section presents an overview of the GCMS-QP2010.
The GCMS-QP2010 instrument is a bench-top type gas chromatograph/mass spectrometer intended for high-precision GC/MS analysis. The instrument enables mass spectrum measurement for qualitative analysis or identification of unknowns and Selected Ion Monitoring (SIM) measurement for quantitation of trace constituents.

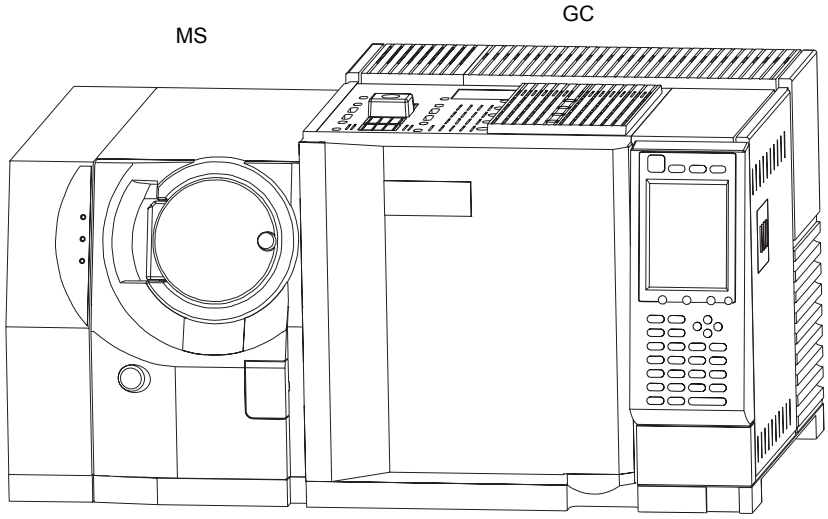


Figure 1.1 GCMS-QP2010

The standard model includes the following components:

- GC-2010 high-performance gas chromatograph
- Vacuum differential pumping* by the turbo molecular pump with the rotary backing pump
- Vacuum gauge for chamber pressure monitoring*
- Direct-coupled GC/MS interface
- Electron Impact (EI) ion source with independent temperature control
- Electron energy/current variable dual filament ion source
- Quadrupole mass filter with pre-rods
- Electron multiplier detector with conversion dynode
- Power source and instrument control circuit

  * For Single TMP models, the vacuum differential pumping becomes a single pumping, and the ion gauge is not included.

GCMSsolution Ver. 2 software is used to control the GCMS-QP2010 and perform data acquisition and post run analysis. The software includes the following functions:

- Control of GCMS-QP2010 and peripherals such as the autosampler, and automatic adjustment of the instrument by autotuning
- QA/QC functions support data precision and quality management.
- Creation of analysis methods
- Scan and SIM data acquisition
- Qualitative processing which includes the display and processing of mass spectrum as well as library searches
- Quantitative processing which includes the creation of compound tables, calibration curves and concentration calculations
- Customizable reports
- Sequential data acquisition/analysis by batch processing

1.2 Features

This section describes the functions and performance features of the GCMS-QP2010.

High Sensitivity
Quadrupole ion focusing GC/MS systems provide the highest sensitivity in their class, significantly broadening the range of applications for GC/MS.

High Mass Range
The instrument achieves a high mass range analysis up to m/z 1024 (for Single TMP models, the analysis can be performed up to m/z 900), significantly broadening the range of applications for GC/MS.

Compact
The GCMS-QP2010 requires minimal space, even with the optional autosampler installed.

Intuitive Operation
Because the GCMSsolution software is intuitive, novices to GC/MS can acquire and analyze data, as well as generate reports in the familiar Windows environment. QA/QC functions provide instrument status and data precision management.

Multi-Compound Analysis
The high sensitivity of the GCMS-QP2010 enables SIM measurements for more compounds. Ions can be monitored simultaneously on a maximum of 64 channels. High sensitivity analysis can be performed on a larger number of compounds because time-division settings may be assigned for up to 128 groups, each with up to 64 channels.

1.3 Component Names

This instrument is composed of a gas chromatograph (GC), mass spectrometer (MS), rotary pump, personal computer system (PC) including monitor and printer, and other options. For the GC-2010 gas chromatography, refer to the separate GC-2010 user manual.

1.3.1 GC/MS Analytical System

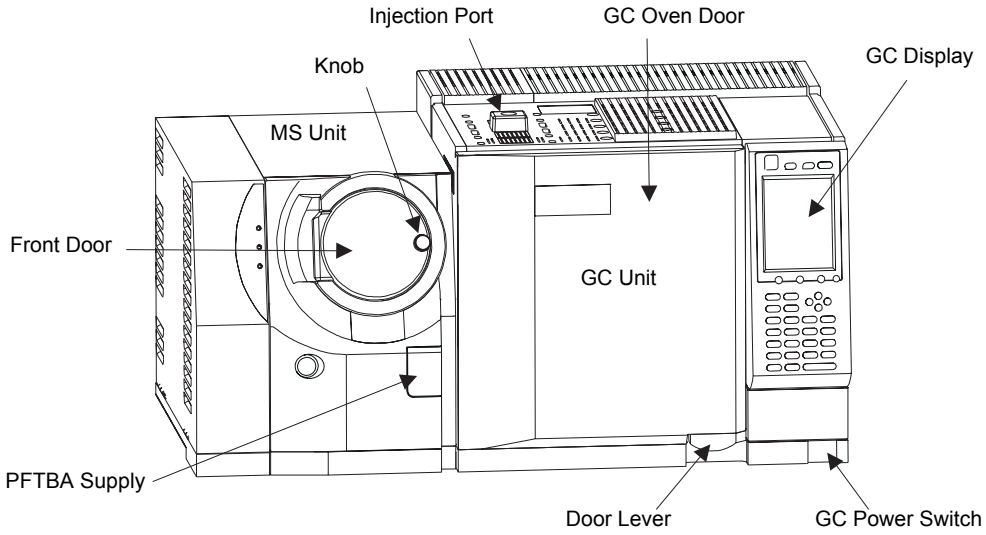


Figure 1.2 GCMS-QP2010 (Front View)

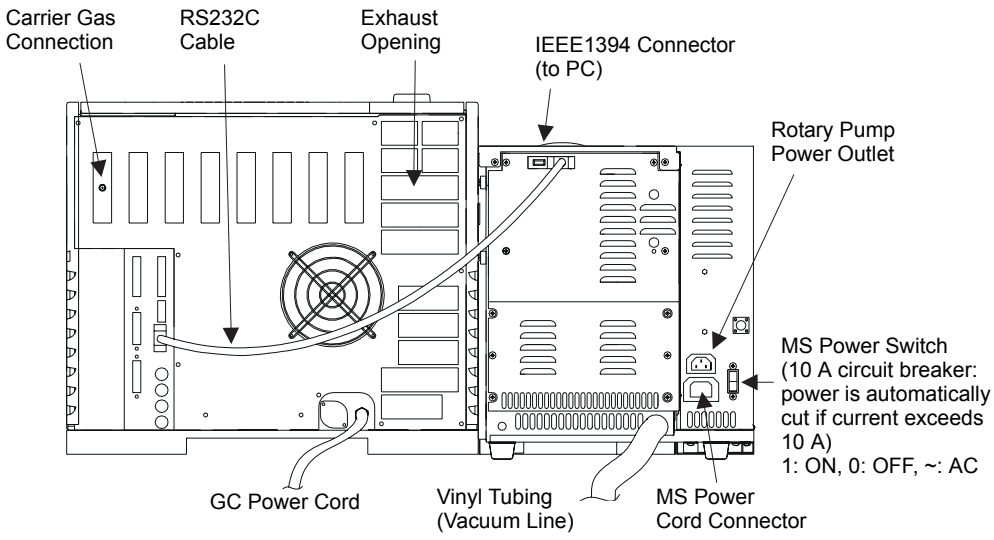
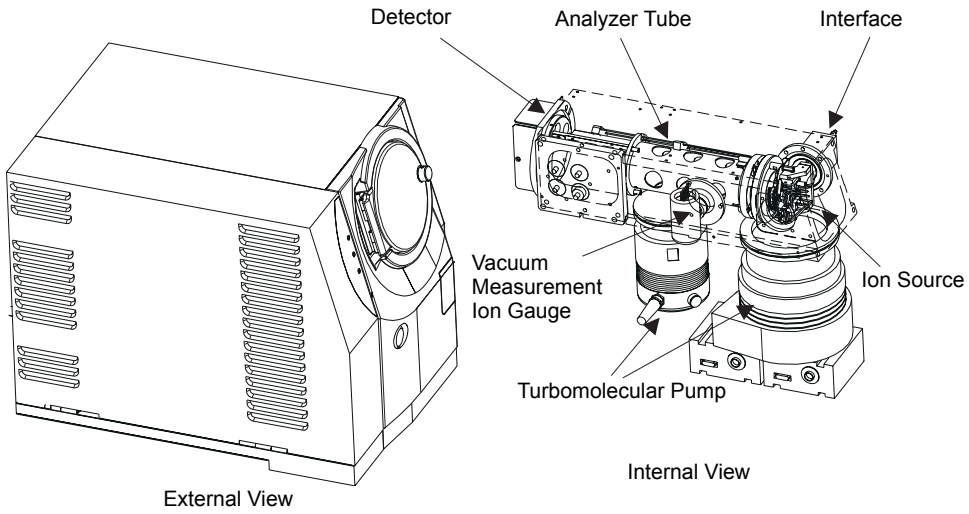


Figure 1.3 GCMS-QP2010 (Rear View)

(Dual TMP model)



(Single TMP model)

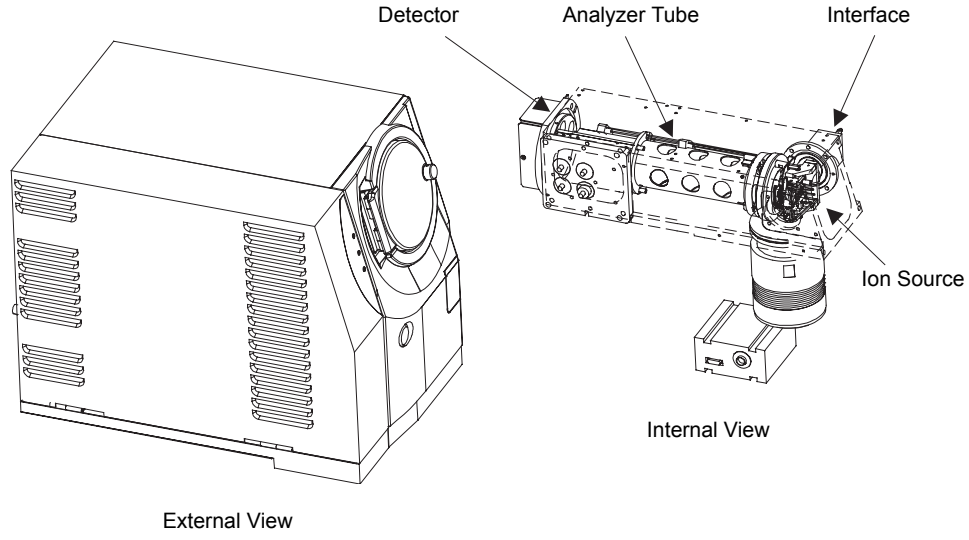


Figure 1.4 Mass Spectrometer

1.3.2 GCMS-QP2010 Ion Source

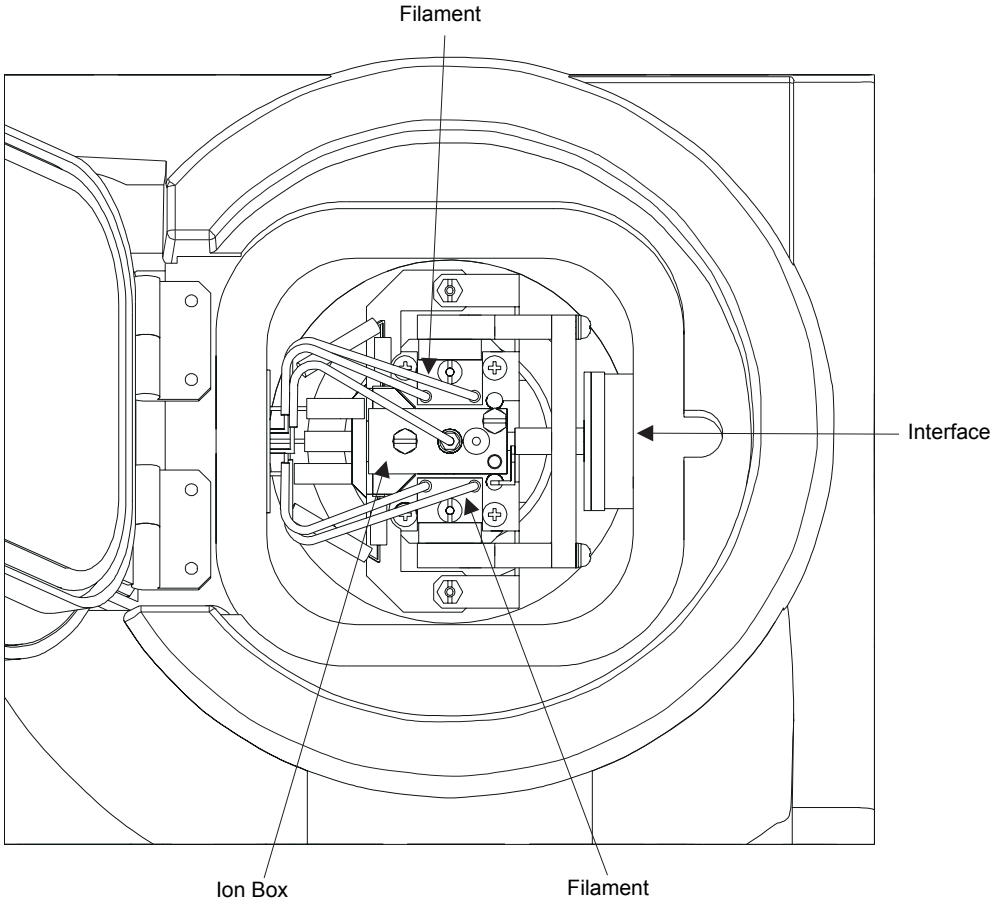


Figure 1.5 Ion Source

1.3.3 Rotary Pump

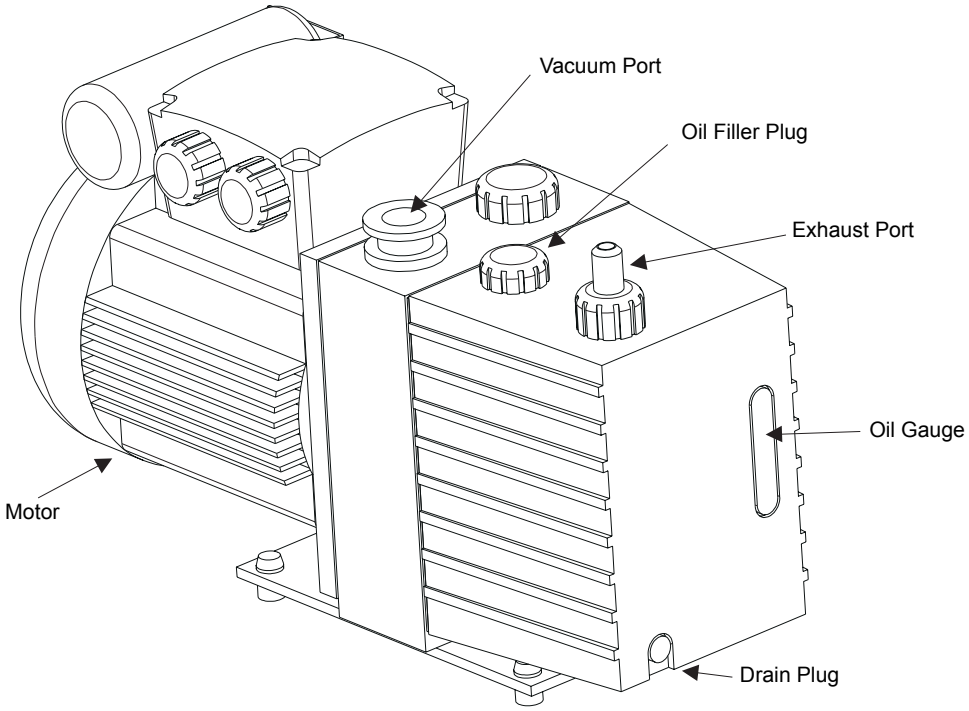


Figure 1.6 Rotary Pump

1.4 Standard Accessories

This section describes the standard accessories that accompany the instrument. Refer to the GC-2010 user manual for the accessories included with the GC-2010 gas chromatograph.

Part Name	Part Number	Qty
Nut, SSNE16 / 012 (5 pcs)	670-11009	1
Ferrule, GVF-004 (10 pcs)	670-15003-03	1
Ferrule, GVF-005 (10 pcs)*	670-15003-04	1
Tweezers, AA	086-16101	1
Screwdriver, Phillips #2	086-11002	1
Ion source attachment jig	225-10194-91	1
Septum (10 pcs)	221-35507-01	1
Wrench (5/16 -1/4)	086-03410	1
Wrench, Hex 3	086-03804	1
Wrench, Hex 5	086-03806	1
Screwdriver, Minus 100	086-10403	1
Screwdriver, #4	086-12011	1
Column mounting jig (to interface)	225-11657-08	1
Column mounting jig (to injector)	225-11657-09	1
Parallel pin	026-11007-02	1
Cable, IEEE1394	225-19050	1
Cable, RS232C	225-19051	1
Gasket	221-48990	1
Case	670-12546	1
Interface cover	225-11674	1
Vinyl tube	016-31331	1
Hose clamp	037-61019	2

* This part is not included with the Single TMP model.

In the case of a Single TMP model, the following parts are also attached.

Part Name	Part Number	Qty
G-type blank nut (2 pcs)	221-35566-92	1
O-ring, 4D P5, 5/PKT, for glass insert attachment	036-11203-84	1
Gasket, AI, column packing (100 pcs)	201-35183	1
Deactivated silica wool, 2 g	221-48600	1
Glass insert, SPL	221-41444-01	1
Glass insert, SLESS	221-48335-01	1
Wrench for glass insert nut	221-46977	1
Injection port cover	221-43597	1
Spanner 10 × 12	086-03011	2
Spanner 6 × 8	086-03003	1

1.5 Software Overview

This section presents an overview of GCMSsolution.

GCMSsolution is Windows software that is used to controls the GCMS-QP2010, autosampler and all other peripheral units. It provides data acquisition, qualitative analyses, quantitative analyses and report creation.

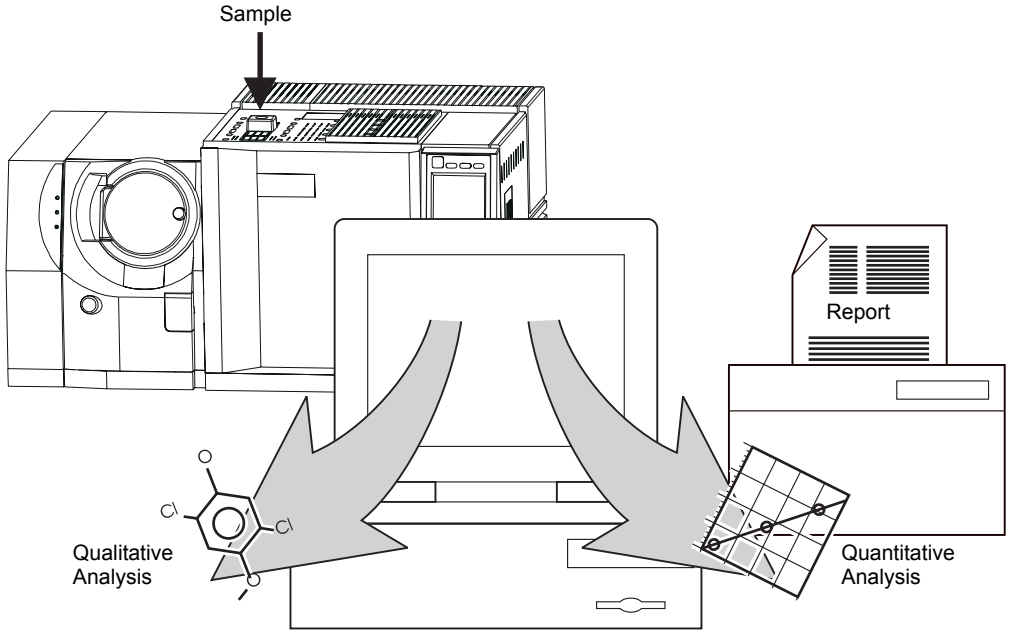


Figure 1.7 Software Functions

The program uses commands that are common to the Windows 2000 and Windows NT 4.0 operating systems. Prior to use, carefully read the user manual of the appropriate operating system. This manual should be read after gaining a basic understanding of Windows operations, such as opening and saving files.

1.6 Software Operation Flow Chart

This flow chart presents the basic order of GCMSsolution operations. The chapters in which the respective procedures are described are listed where appropriate.

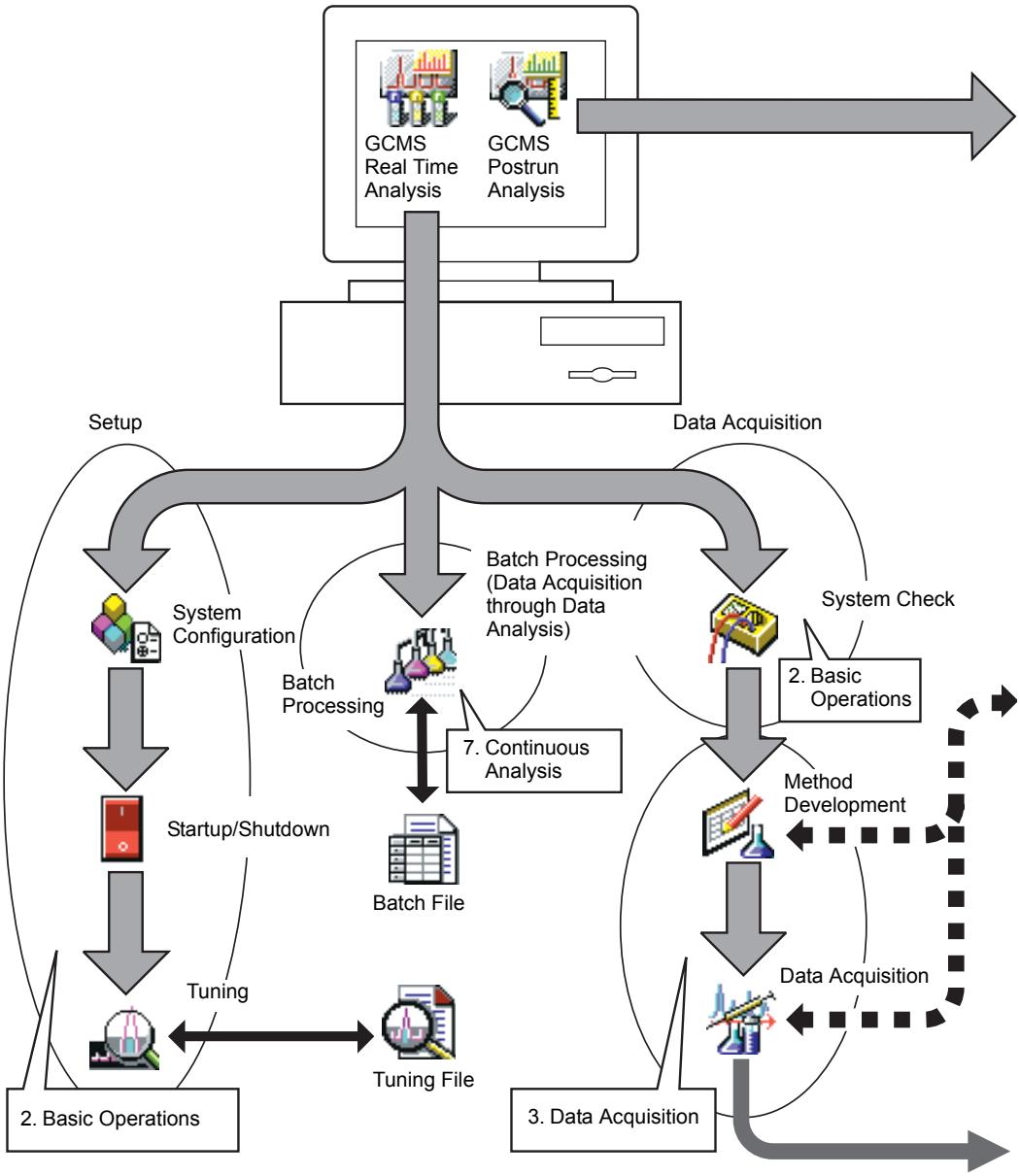


Figure 1.8 Software Operation Flow Chart (Part 1)

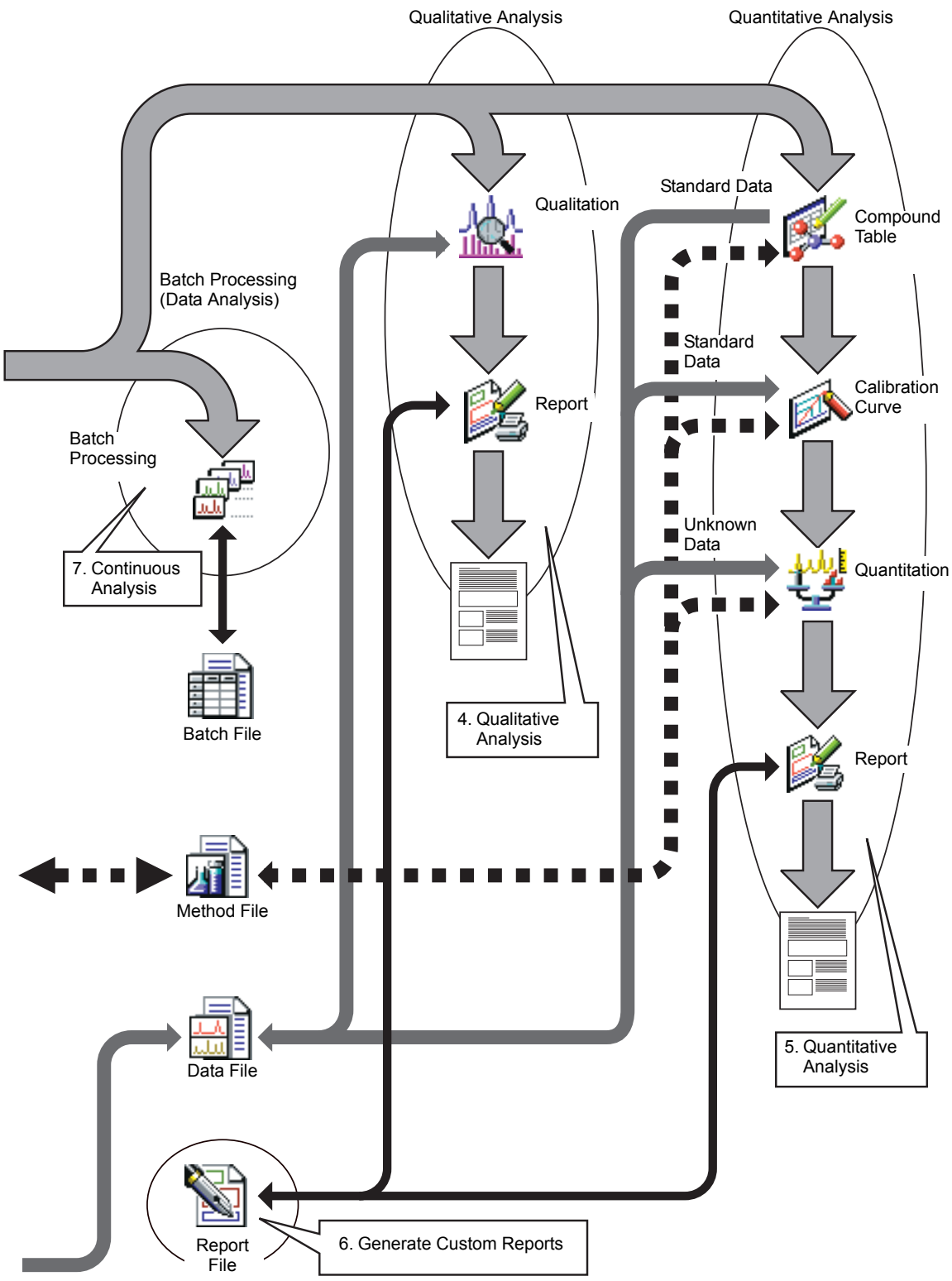


Figure 1.9 Software Operation Flowchart (Part 2)

2 Basic Operation

2.1 Introduction

This section describes the following basic operational procedures of GCMSsolution:

- GC Basics
- Gas Requirements
- Instrument Startup and Shutdown
- Daily Startup and Shutdown
- Column Replacement
- System Configuration
- System Check and Tuning
- Common Operations

An understanding of these topics is crucial for operating the GCMS-QP2010 instrument and GCMSsolution software.

2.2 GC Basics

For specific information about the GC-2010 gas chromatograph, consult its accompanying user manual (P/N 221-40406). Note that the standard procedures for operating the GC-2010 may differ from those described below.

2.2.1 Instrument Parameters

The GCMS-QP2010 instrument parameters, including GC temperature and pressure parameters, may be set from the PC. The parameters are saved in a file referred to as the "method file." Method files may be stored on the hard drive and the saved parameters loaded into the software. For more information about method files, refer to Section 2.9.1 "Managing Files", page 60.

GC-2010 parameters may be set directly from the keypad or from a method file on the PC.

2.2.2 Injection Port Description

With a GC/MS system, air must be prevented from leaking into the carrier gas. Ensuring carrier gas purity will:

1. Prevent capillary column deterioration.
2. Prolong the life of the MS filament and ion source.
3. Minimize the background noise in the analyzer.

To prevent air from leaking into the carrier gas, the GCMS-QP2010 seals the standard injection port with a gold (Au) gasket, as shown in the diagram below.

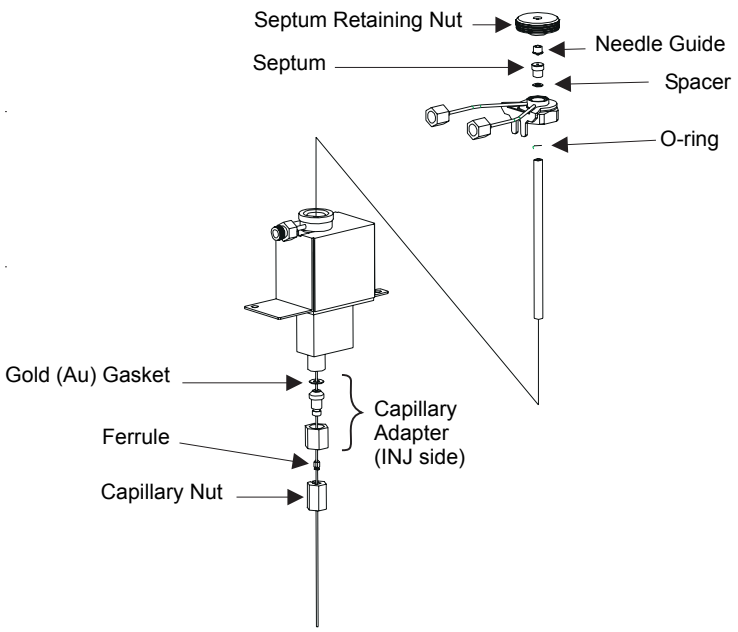


Figure 2.1 Injection Port

2.3 Gas Requirements

This section describes the carrier gas used by this system. The specifications presented below must be followed to promote safety and maintain instrument performance.

Carrier gas: Helium

Supply Pressure: 300 - 980 kPa

Purity: 99.995 % or greater

- The pressure and flow rate setting range on the GC varies according to the supply pressure. Normally a supply pressure of 700 - 800 kPa is necessary.
- Some optional accessories may require a gas other than the one specified above. Consult the user manual accompanying the accessory for more information.
- Some applications, such as agrochemical analysis, may require helium of a higher purity (99.999 - 99.9999 %).

Note

Any air leakage into the gas supply lines will degrade the purity of the gas and may affect system performance. The helium supplied to the GC must meet or exceed the given purity specification. Using a gas of lesser purity may deteriorate system performance.

2.4 Instrument Startup and Shutdown

This section explains how to start up and shut down an installed instrument.

2.4.1 Starting the Instrument

1. Turn on the carrier gas.

(1) After verifying that the carrier gas is helium with a purity of at least 99.995 %, make sure that the carrier gas lines are connected as shown in the diagram below.

(2) Open the main valve of the carrier gas cylinder.

(3) Ensure that the supply pressure, or the pressure at GC carrier gas inlet, is 300 - 980 kPa (about 3 - 9.8 kgf/cm^2).

Note

When changing gas cylinders, make sure that there are no leaks from the connection at the gas cylinder.

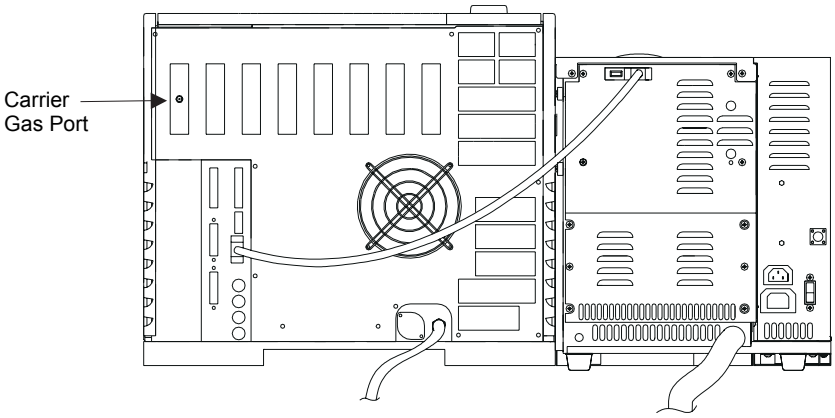


Figure 2.2 Carrier Gas Inlet

2. Inspect the injection port glass insert using the following procedure to determine whether the glass insert is appropriate for the application.

(1) Hold the septum nut and remove the glass insert nut of the injection port. Lift the septum nut straight up and remove it.

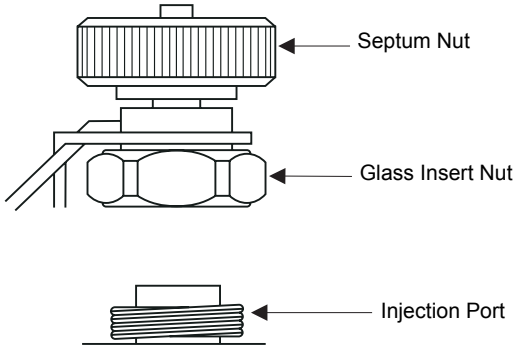


Figure 2.3 Injection Port Nut Assembly

Caution

It is important to hold the septum nut while removing the glass insert nut. If you remove the glass insert nut without holding the septum nut, it is pulled by the tube. The septum nut will hit the glass insert and break it.

(2) Remove the glass insert with forceps, and verify that it is appropriate for the application. Refer to the figure below. Check the silica wool to make sure that it is clean and positioned properly.

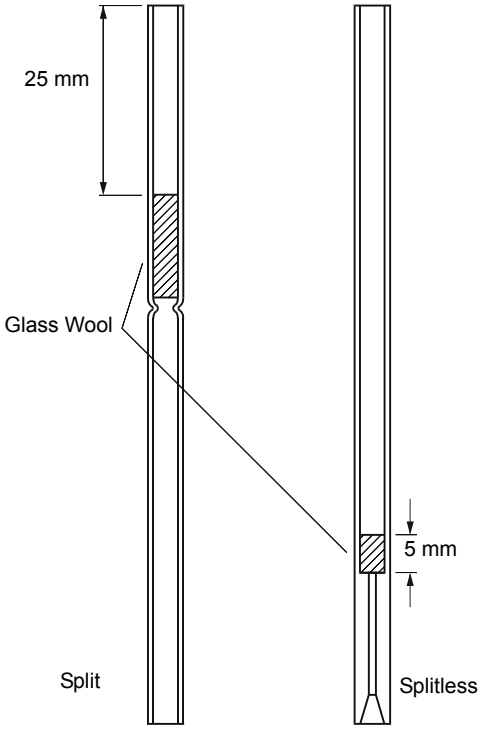


Figure 2.4 Packing Glass Inserts

(3) If the silica wool is contaminated, remove the wool, and clean or replace it. Pack an appropriate amount of clean silica wool at the specified location. For split injection this is about 10 mg and for splitless about 2 mg.

(4) Install the glass insert and graphite ferrule as described below. Refer to the figure of the upper injection port assembly as necessary.
   a) Temporarily slide the O-ring onto the glass insert so that it is about 4 mm from the top, as shown in the diagram below. Guide the glass insert into the injection port until it touches the bottom. The O-ring will be about 3 mm from the top of the insert when properly positioned.
   b) Position the injection port nut assembly on top of the glass insert.
   c) Securely tighten the nut with the provided hex wrench.

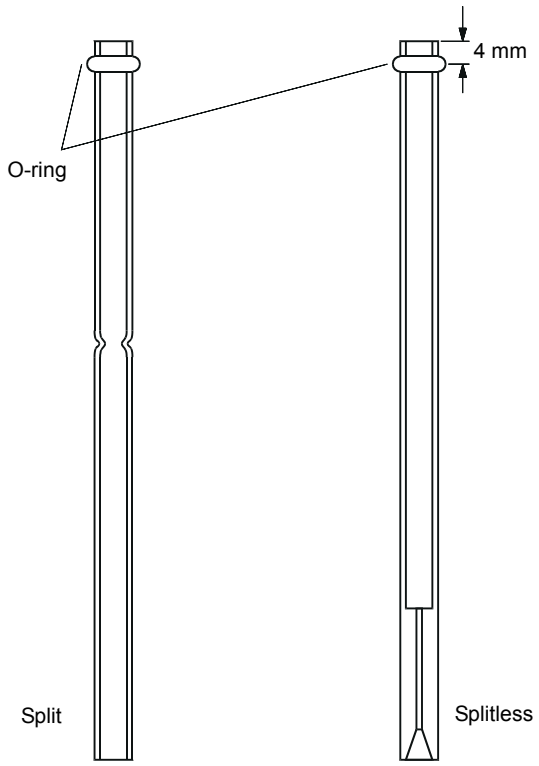


Figure 2.5 Placing the O-ring

3. Install the column holder and capillary column.

Attaching the Column Hanger

Attach the column hanger to the connecting holes as follows:

(1) Squeeze the tabs at the top of the hanger so the ends of the hanger can be inserted into the connecting holes. The standard column mounting position is on the back side of the hanger. When connecting two columns, mount one in front and the other in back.

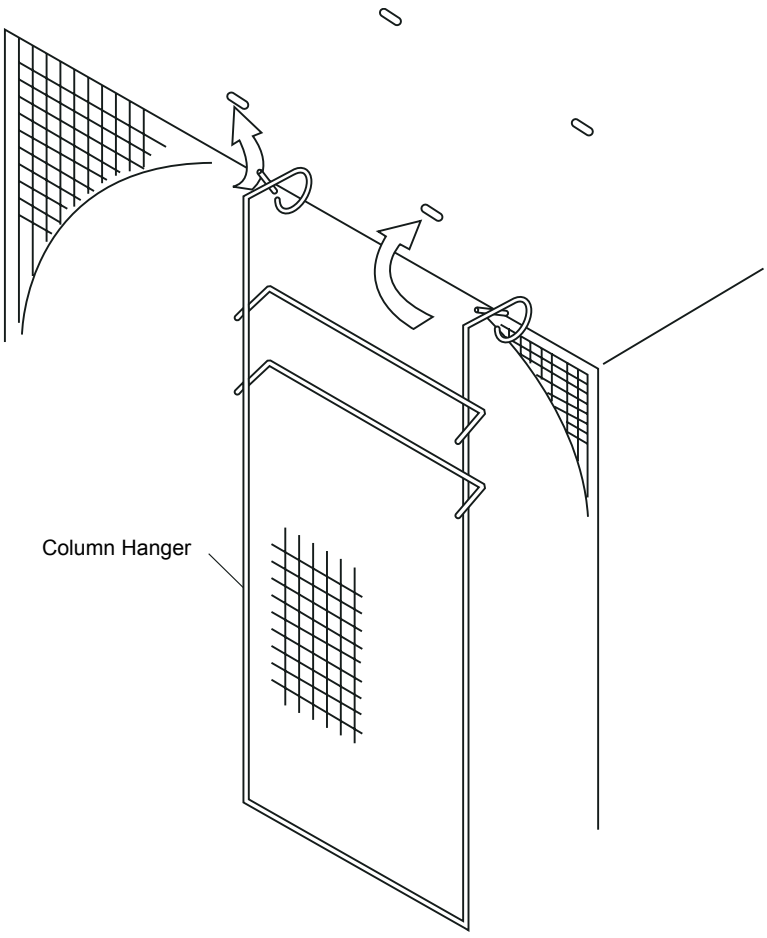


Figure 2.6 Connecting the Column to the Column Hanger

Connecting the Capillary Column to the Injection Port

(1) Push the capillary column through the Vespel ferrule and nut. Refer to Figure 2.7 "Using the Column Fitting Jig".

(2) Insert the end of the capillary column into the column fitting jig (P/N 225-11657-09), so that the capillary column protrudes about 1 cm from the end of the jig as indicated in Figure 2.7 "Using the Column Fitting Jig" Secure it by tightening the nut. Cut the protruding end of capillary column.

(3) Mark the capillary column beneath the tightened nut with tape as shown in Figure 2.8 "Marking the Column with Tape".
(4) Remove the capillary column from the jig without moving the tape, and wipe the capillary column with acetone. Insert the column into the injection port and secure it by hand tightening the nut. If the ferrule is new, use the wrench to turn the nut an additional full turn. If the ferrule has been previously used, use the wrench to tighten the nut by turning it 20 to 40 degrees.
(5) Remove the tape used to mark the column.

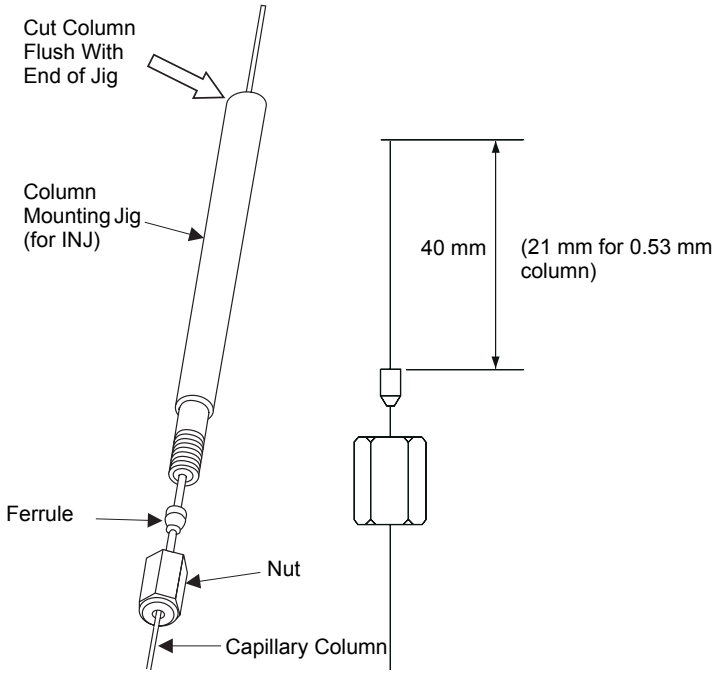


Figure 2.7 Using the Column Fitting Jig

Part Name	Part Number
Vespel ferrule	670-15003-03 (for 0.25 mm column)
	670-15003-04 (for 0.32 mm column)
	670-15003-07 (for 0.53 mm column)
Column mounting jig	225-11657-09
Nut	670-11009
Capillary column cutter	221-50595-91 (option)

Note

A graphite ferrule is crimped by the jig, so that it is attached to the column, whereas a Vespel ferrule is not crimped and will slide on the column. To ensure proper positioning of the column, mark the column beneath the Vespel ferrule and nut with tape. Ensure that the tape is removed after installation.

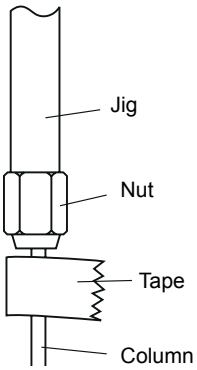


Figure 2.8 Marking the Column with Tape

Connecting the capillary column to the MS

(1) Push the capillary column through the Vespel ferrule and nut. Refer to Figure 2.9 "Using the Column Mounting Jig".

(2) Insert the end of the capillary column into the column fitting jig (P/N 225-11657-08), so that the capillary column protrudes about 1 cm from the end of the jig as indicated in Figure 2.10 "Connecting the Capillary Column to the MS". Secure it by tightening the nut. Cut the protruding end of capillary column.

(3) Mark the capillary column beneath the tightened nut with tape as shown in Figure 2.8 "Marking the Column with Tape".

(4) Remove the capillary column from the jig without moving the tape, and wipe the capillary column with acetone. Insert the column into the MS interface and secure it by hand tightening the nut. If the ferrule is new, use the wrench to turn the nut an additional full turn. If the ferrule has been previously used, use the wrench to tighten the nut by turning it 20 to 40 degrees.

(5) Remove the tape used to mark the column.

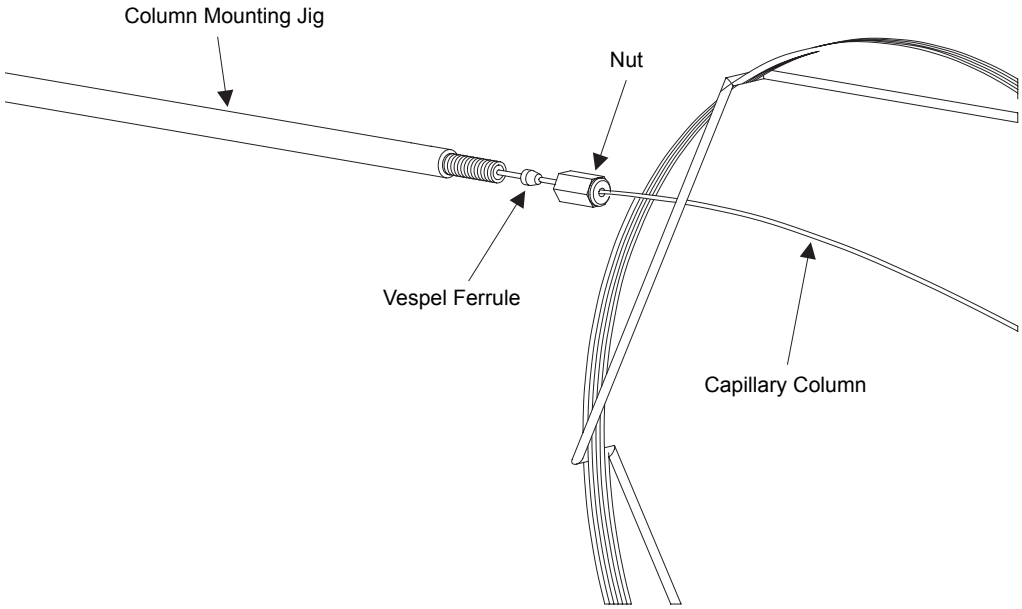


Figure 2.9 Using the Column Mounting Jig

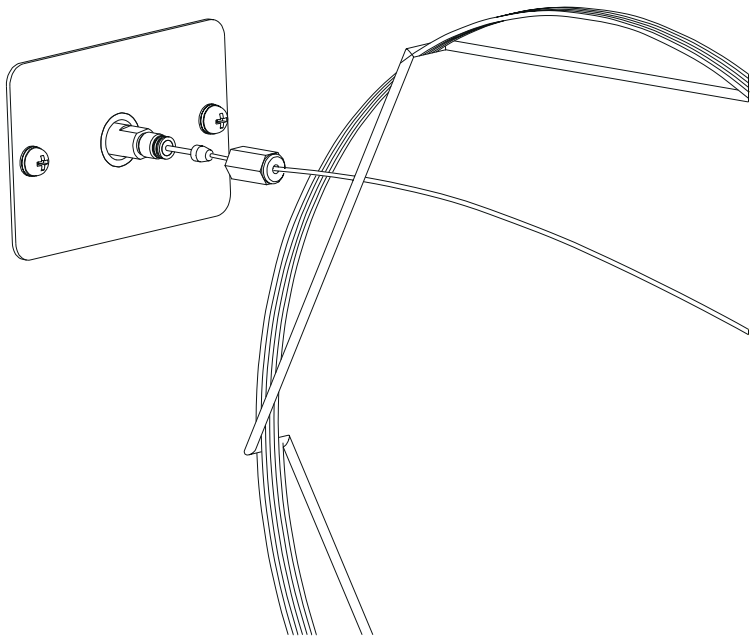


Figure 2.10 Connecting the Capillary Column to the MS

Note

To prevent background noise, always use a Vespel ferrule to connect the column to the MS. A graphite ferrule is crimped by the jig, so that it is attached to the column, whereas the Vespel ferrule is not crimped and will slide on the column. To ensure proper positioning of the column, mark the column beneath the Vespel ferrule and nut with tape. Ensure that the tape is removed after installation.

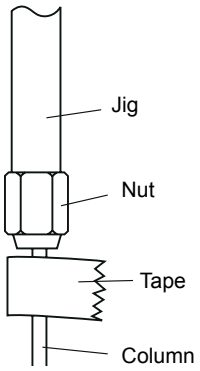


Figure 2.11 Marking the Column with Tape

4. Turn on the instrument.

(1) Ensure that breaker controlling the instrument power supply is on, and turn on the GC.

(2) Turn on the computer, monitor and printer, and start Windows.

(3) Turn on the MS. The power LED in the upper left corner of the MS will illuminate.

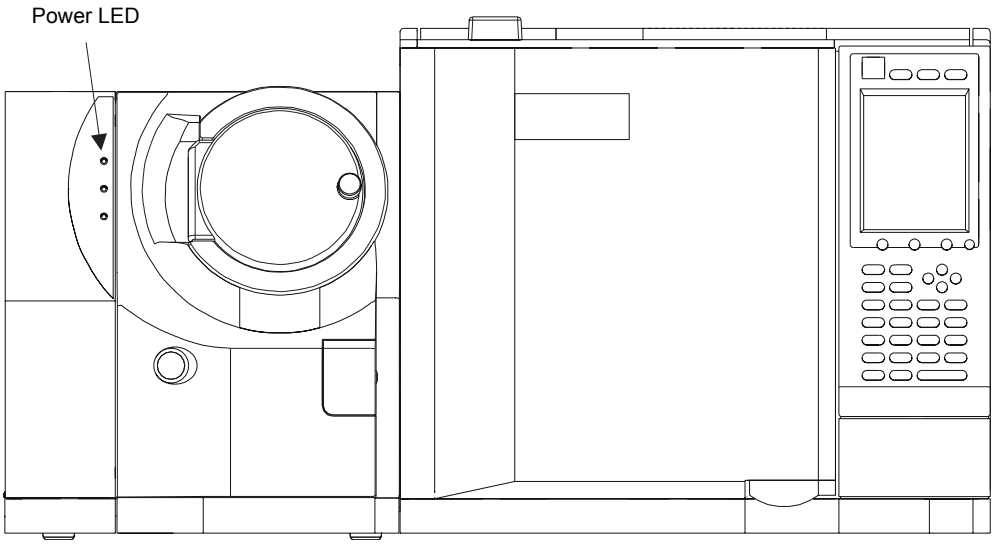


Figure 2.12 Starting the MS

5. Start GCMSsolution.

(1) Double-click the **GCMS Real Time Analysis** icon. The "Login" dialog box is displayed.

Figure 2.13 "Login" dialog box

(2) Enter your user name and password. The first time that the software is accessed, use the default user name "Admin" and leave the password blank.
After entering the necessary information, click the **OK** button.
The GCMSsolution software starts up, and the "GCMS Real Time Analysis" window is displayed.

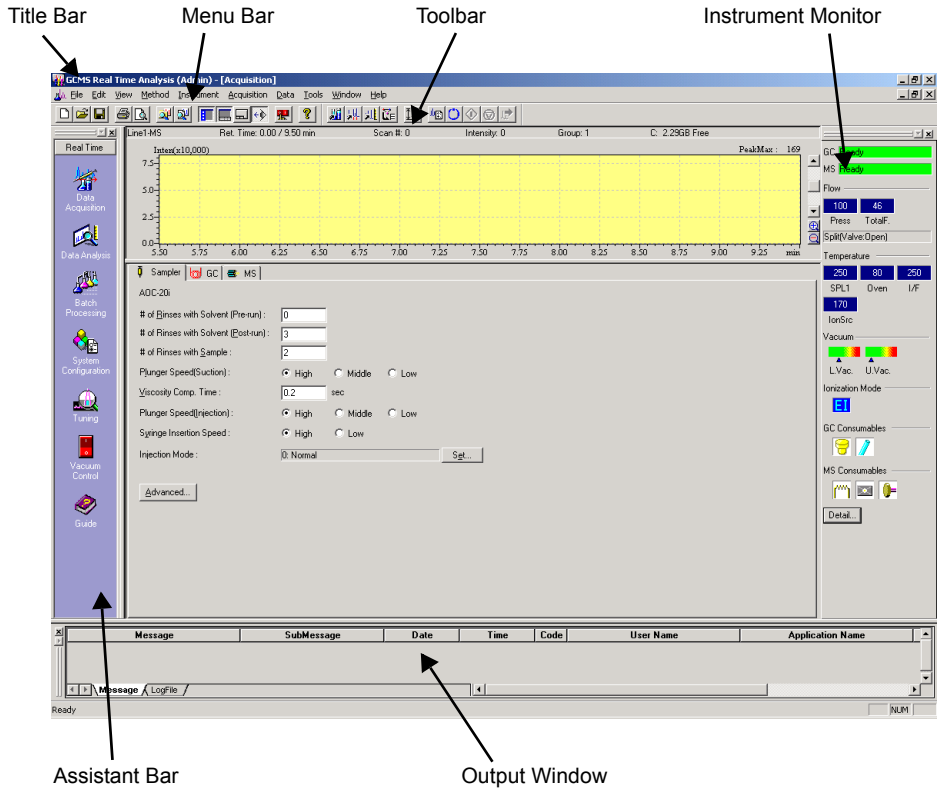


Figure 2.14 "GCMS Real Time Analysis" Window

6. Start the vacuum system.

Caution

If the instrument has been shut down for an extended period of time and the ambient temperature is low, the rotary pump oil may become very viscous. When the oil viscosity is too high and the rotary pump is operated, an excessive load is placed on the rotary pump motor. The breaker for the main power supply (on back of the instrument, see Section 1.3.1 "GC/MS Analytical System", page 3) may trip, shutting down the instrument.

When the instrument has been turned off and maintained in a cold environment, increase the ambient temperature and start up the instrument after the rotary pump temperature is at least as high as the minimum temperature specification (18 °C).

Caution

Verify that the knob on the MS front panel door is tight before starting the vacuum system.

Caution

Do not tighten the knob on the MS front panel door after starting the vacuum system. Otherwise, it may be impossible to loosen the knob after the instrument is shut down.

(1) Click the **Vacuum Control** icon in the Assistant Bar. The "Vacuum Control" dialog box is displayed.

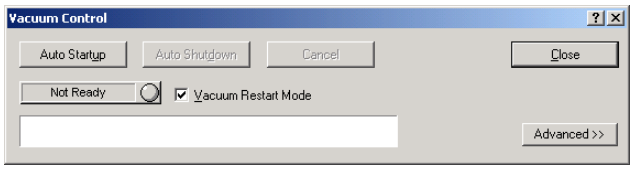


Figure 2.15 "Vacuum Control" Dialog Box

(2) Click the **Auto Startup** button.
The Startup indicator flashes, the progress bar is displayed, and the vacuum system startup sequence initiates. The various components start up in sequence, as noted in the progress bar, and when the vacuum system is ready for operation, "Completed." is displayed.

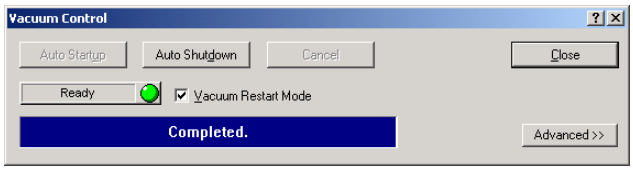


Figure 2.16 Vacuum Startup Completed

(3) Close the "Vacuum Control" dialog box.

2.4.2 Shutting Down the Instrument

1. Shut down the vacuum system.

(1) Click the Assistant Bar **Vacuum Control** icon to open the "Vacuum Control" dialog box.

(2) Click the **Auto Shutdown** button.
The Shutdown indicator flashes, the progress bar is displayed, and the vacuum system shutdown sequence initiates. The various components shut down in sequence, as noted in the progress bar, and when the vacuum system shutdown is completed, "Completed." is displayed.

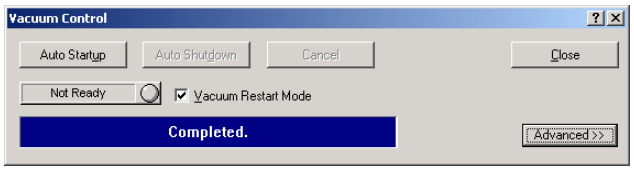


Figure 2.17 Vacuum System Shutdown Completed

(3) Close the "Vacuum Control" dialog box.

2. Exit GCMSsolution.

(1) Select Exit from the File menu. A confirmation message is displayed to verify exiting the program.

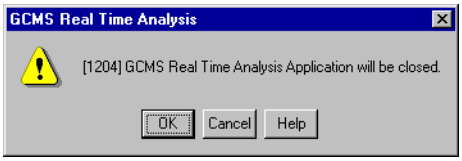


Figure 2.18 Exit GCMSsolution Confirmation

(2) Click the **OK** button to exit GCMSsolution.

3. Turn off the power supply and carrier gas.

(1) Turn off the computer, monitor and printer.
(2) Turn off the GC.
(3) Turn off the MS.
(4) Close the main valve of the carrier gas cylinder.

2.5 Daily Startup and Shutdown

When a GC/MS system is used daily, the GC and MS components, including the vacuum system, are not shut down and remain in operation. Usually, the software is exited and the computer shut down. This section describes how to shut down and start up the computer and software while the rest of the system remains running.

2.5.1 Daily Shutdown

When the instrument is used daily, the vacuum system should be left running after each use rather than shutting down and restarting it so that the proper analysis conditions can be met more quickly. This section outlines the daily shutdown procedure when the vacuum system is left running.

1. Select Daily Shutdown from the Tools menu to open the "Daily Shutdown" dialog box.

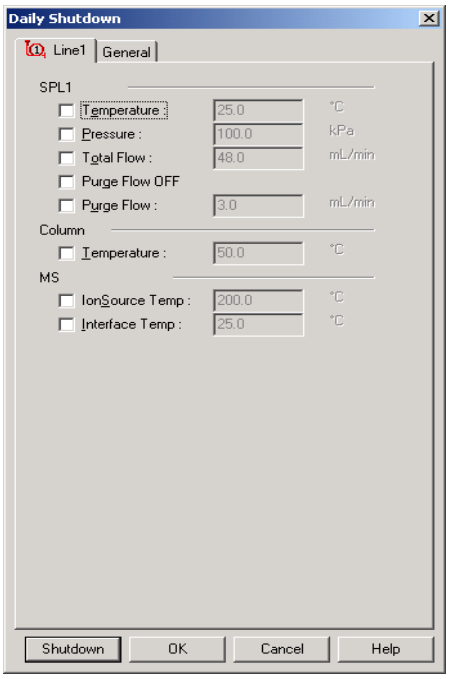


Figure 2.19 "Daily Shutdown" Dialog Box

2. Enter the desired settings for the equipment in use on the Line 1 and General tab, etc.

3. Click the **Shutdown** button. The "Daily Shutdown" dialog box closes. The instrument will remain running according to the parameters entered in this window.

4. Exit Windows.

5. Turn off the computer, monitor and printer.

Note

Do not turn off the GC or MS.
Do not turn off the main power supply.
Do not change the GC column inlet pressure to 0.

2.5.2 Daily Startup

Follow the procedure below when the vacuum system is already running, as when the daily shutdown procedure has been performed.

1. Turn on the computer, monitor and printer, and start Windows.

2. Double-click the **GCMS Real Time Analysis** icon. The "Login" dialog box is displayed.

Figure 2.20 "Login" Dialog Box

3. Enter your user name and password, and click **OK**. The GCMSsolution software starts up, and the "GCMS Real Time Analysis" window is displayed.

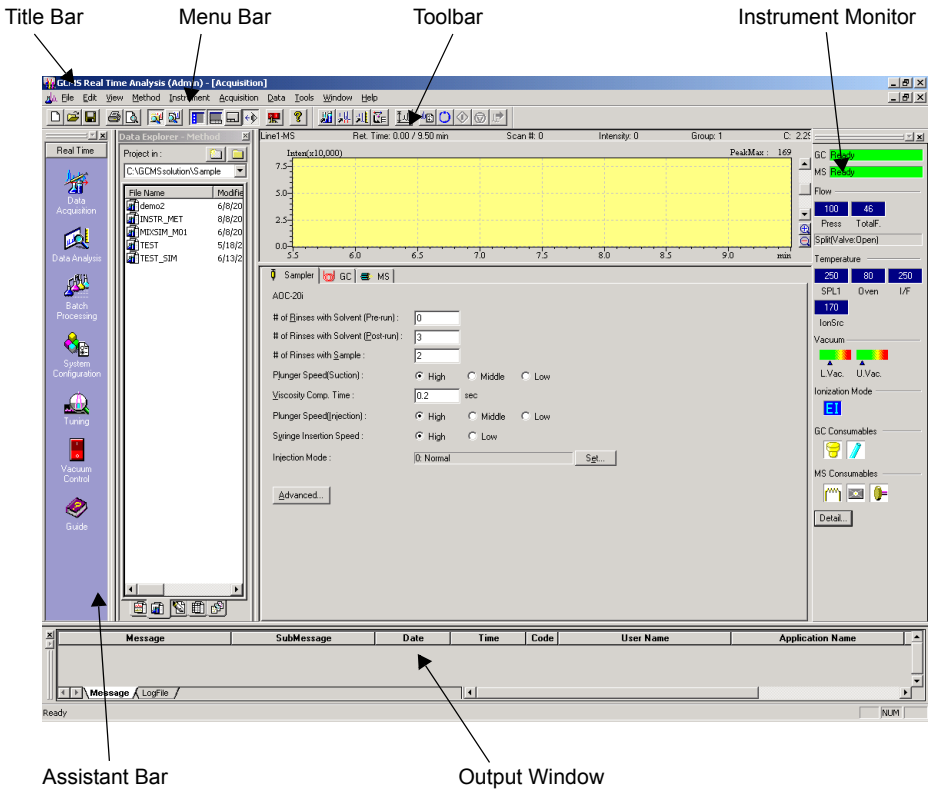


Figure 2.21 "GCMS Real Time Analysis" Window

2.6 Column Replacement

This section explains column replacement when column performance has deteriorated or with a column more appropriate for the application. If the column is not replaced correctly, a vacuum leak may result.

2.6.1 Shutting Down the Vacuum System

1. Click the Assistant Bar **Vacuum Control** icon to open the "Vacuum Control" dialog box.

2. Click the **Auto Shutdown** button.
   The Shutdown indicator flashes, the progress bar is displayed, and the vacuum system shutdown sequence initiates. The various components shut down in sequence, as noted in the progress bar, and when the vacuum system shutdown is completed, "Completed." is displayed.

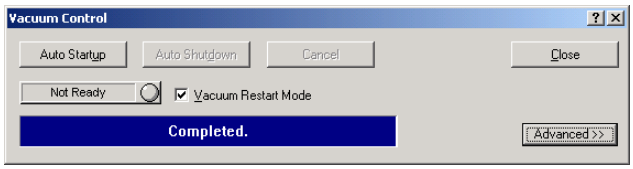


Figure 2.22 Vacuum System Shutdown Completed

3. Close the "Vacuum Control" dialog box.

Caution

If the front door of the vacuum housing will be opened after the vacuum system is shut down, loosen the knob on the front door with one turn to the left.

2.6.2 Changing the Column

1. Remove the column that is currently installed.
Loosen the nuts at the injector and MS connections, and remove the capillary column. If the Vespel ferrule and nut from the MS end are still on the capillary column, the column may be stored with the ferrule and nut attached.

The Vespel ferrules and nuts required to install the capillary column are considered consumable parts and should be stocked in sufficient quantities. Refer to Section D.1 "Consumable Parts List", page 333.

2. Install the new column.

Connecting the Capillary Column to the Injection Port

(1) Push the capillary column through the Vespel ferrule and nut. Refer to Figure 2.23 "Using the Column Fitting Jig".
(2) Insert the end of the capillary column into the column fitting jig (P/N 225-11657-09), so that the capillary column protrudes about 1 cm from the end of the jig as indicated in Figure 2.23 "Using the Column Fitting Jig" Secure it by tightening the nut. Cut the protruding end of capillary column.
(3) Mark the capillary column beneath the tightened nut with tape as shown in Figure 2.24 "Marking the Column with Tape".
(4) Remove the capillary column from the jig without moving the tape, and wipe the capillary column with acetone. Insert the column into the injection port and secure it by hand tightening the nut. If the ferrule is new, use the wrench to turn the nut an additional full turn. If the ferrule has been previously used, use the wrench to tighten the nut by turning it 20 to 40 degrees.
(5) Remove the tape used to mark the column.

Part Name	Part Number
Vespel ferrule	670-15003-03 (for 0.25 mm column)
	670-15003-04 (for 0.32 mm column)
	670-15003-07 (for 0.53 mm column)
Column mounting jig	225-11657-09
Nut	670-11009
Capillary column cutter	221-50595-91 (option)

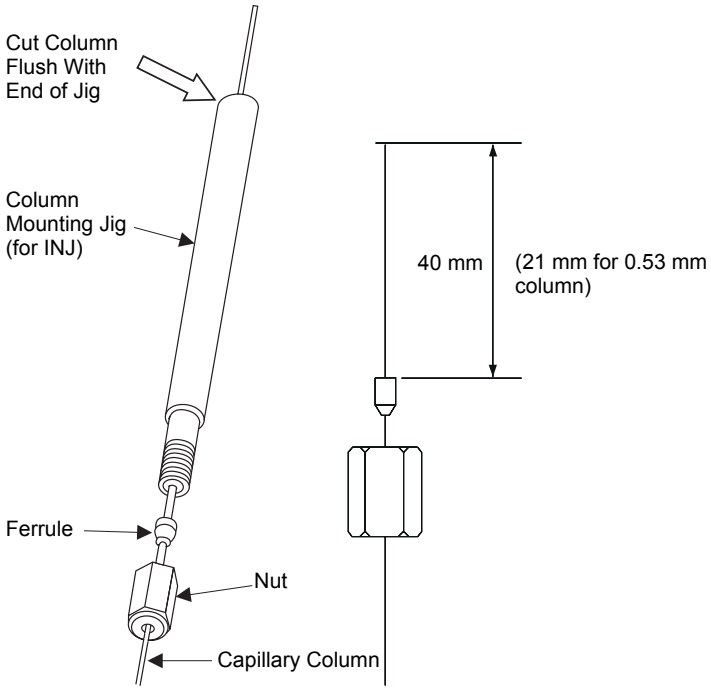


Figure 2.23 Using the Column Fitting Jig

Note

A graphite ferrule is crimped by the jig, so that it is attached to the column, whereas a Vespel ferrule is not crimped and will slide on the column. To ensure proper positioning of the column, mark the column beneath the Vespel ferrule and nut with tape.

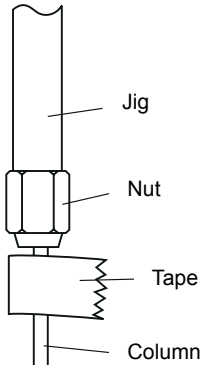


Figure 2.24 Marking the Column with Tape

Connecting the capillary column to the MS

(1) Push the capillary column through the Vespel ferrule and nut. Refer to Figure 2.25 "Using the Column Mounting Jig".

(2) Insert the end of the capillary column into the column fitting jig (P/N 225-11657-08), so that the capillary column protrudes about 1 cm from the end of the jig as indicated in Figure 2.26 "Connecting the Capillary Column to the MS". Secure it by tightening the nut. Cut the protruding end of capillary column.

(3) Mark the capillary column beneath the tightened nut with tape as shown in Figure 2.24 "Marking the Column with Tape".

(4) Remove the capillary column from the jig without moving the tape, and wipe the capillary column with acetone. Insert the column into the MS interface and secure it by hand tightening the nut. If the ferrule is new, use the wrench to turn the nut an additional full turn. If the ferrule has been previously used, use the wrench to tighten the nut by turning 20 to 40 degrees.

(5) Remove the tape used to mark the column.

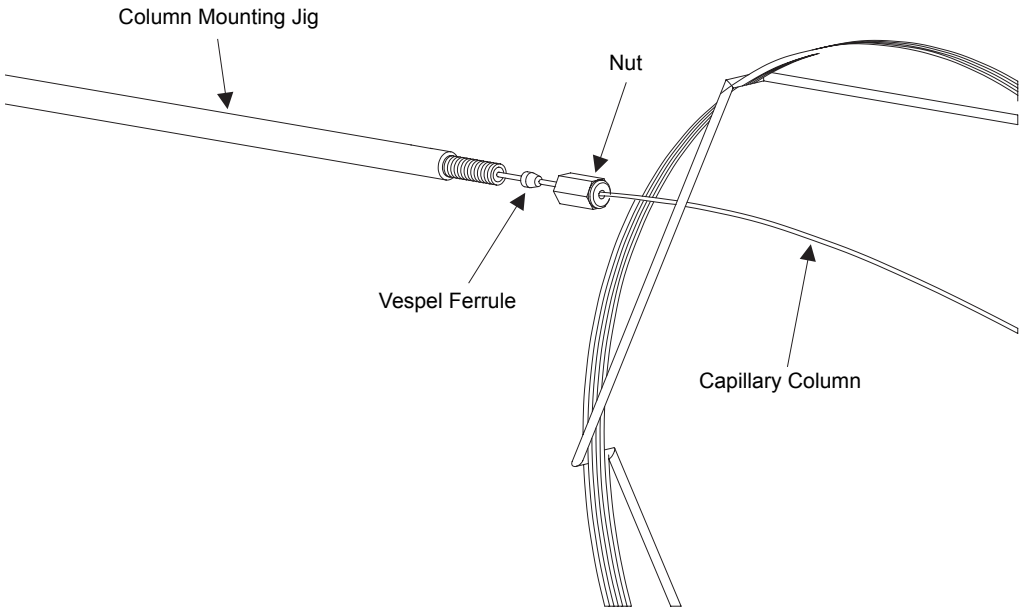


Figure 2.25 Using the Column Mounting Jig

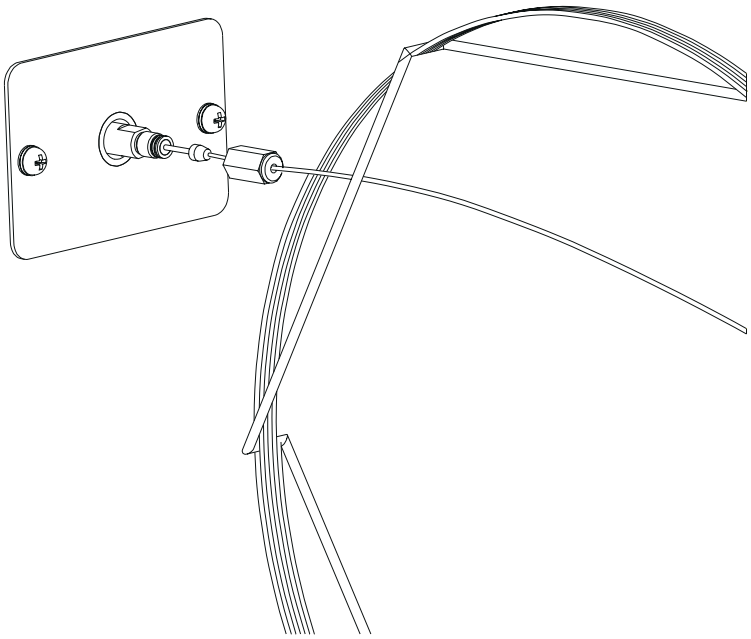


Figure 2.26 Connecting the Capillary Column to the MS

Note

To prevent background noise, always use a Vespel ferrule to connect the column to the MS. A graphite ferrule is crimped by the jig, so that it is attached to the column, whereas the Vespel ferrule is not crimped and will slide on the column. To ensure proper positioning of the column, mark the column beneath the Vespel ferrule and nut with tape.

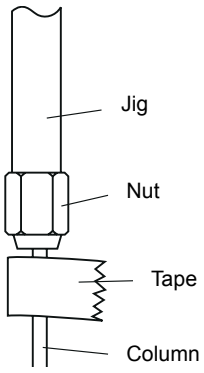


Figure 2.27 Marking the Column with Tape

Note

A driftpin is provided to remove the ferrule lodged inside the mounting nut (SSNE 16/012) when the nut is re-used. In order to reduce wear on the interface screw, only reuse it two or three times.

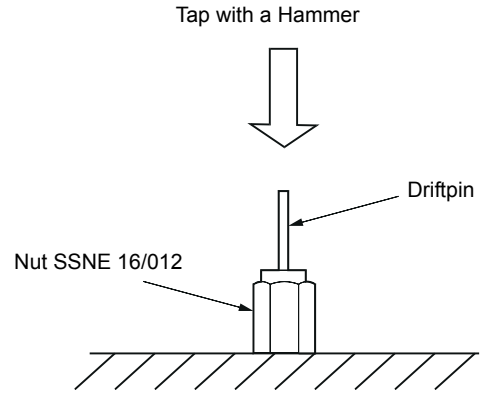


Figure 2.28 Using the Driftpin to Remove Ferrule

3. Turn on the instrument.

(1) Ensure that the breaker controlling the instrument power supply is on, and turn on the GC.

(2) Turn on the computer, monitor and printer, and start Windows.

(3) Turn on the MS. The power LED in the upper left corner of the MS will light.

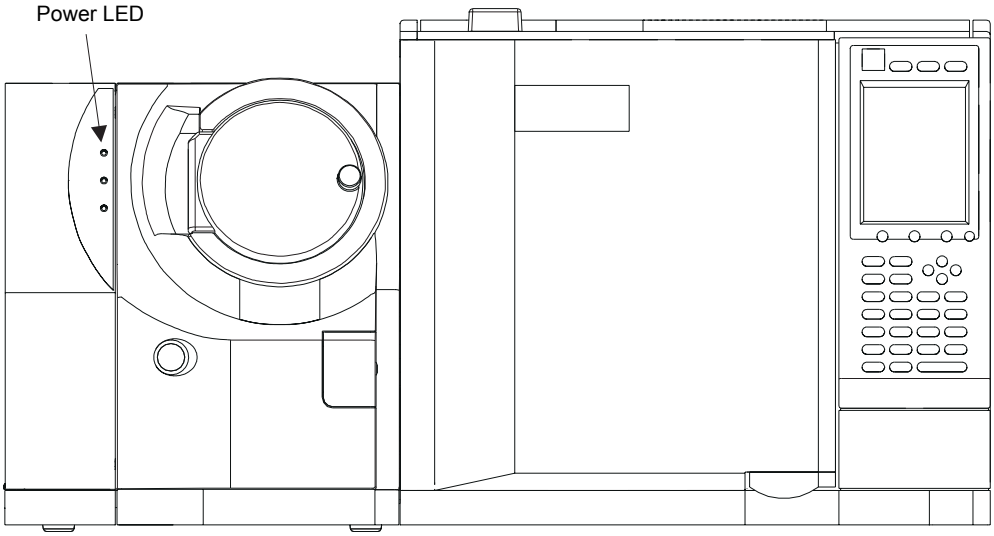


Figure 2.29 Starting the Instrument

4. Start GCMSsolution.

(1) Double-click the **GCMS Real Time Analysis** icon. The "Login" dialog box is displayed.

Figure 2.30 "Login" Dialog Box

(2) Enter your user name and password. The first time that the software is accessed, use the default user name "Admin" and leave the password blank.
Click the **OK** button.
The GCMSsolutions2 software starts up, and the "GCMS Real Time Analysis" window is displayed.

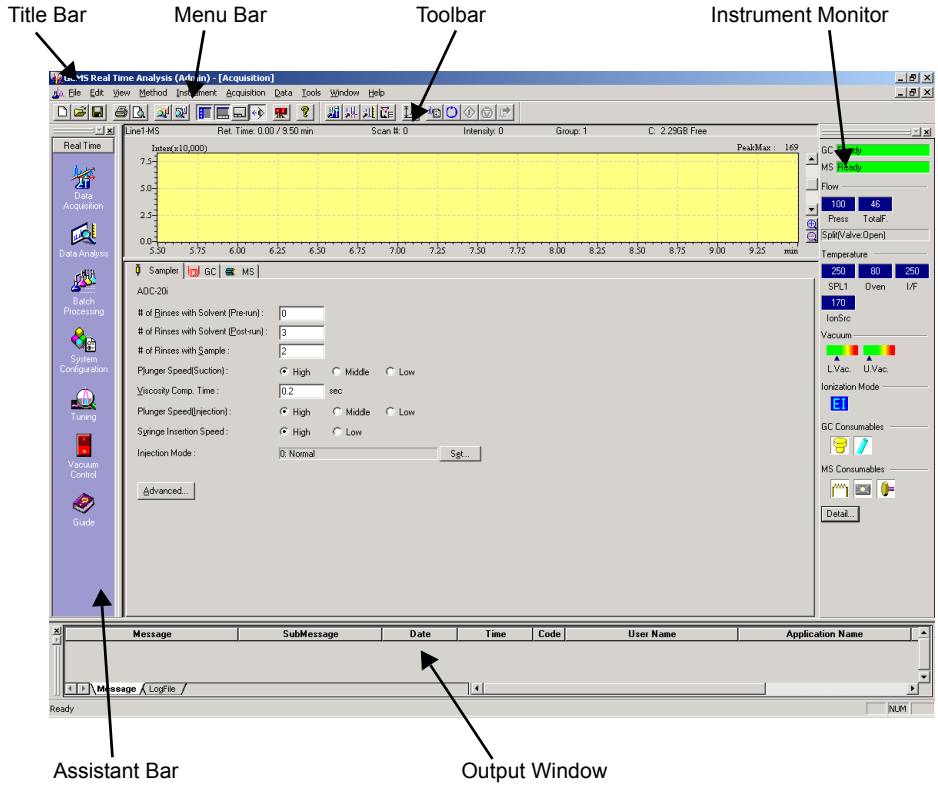


Figure 2.31 "GCMS Real Time Analysis" Window

5. Start the vacuum system.

Caution

If the instrument has been shut down for an extended period of time and the ambient temperature is low, the rotary pump oil may become very viscous. When the oil viscosity is too high and the rotary pump is operated, an excessive load is placed on the rotary pump motor. The breaker for the main power supply (on the back of the instrument, Section 1.3.1 "GC/MS Analytical System", page 3) may trip and shut down the instrument.
When the instrument has been turned off and kept in a cold environment, increase the ambient temperature and start up the instrument after the rotary pump temperature is at least as high as the minimum temperature specification (18 °C).

Caution

Before starting up the vacuum system, confirm that the knob of the front door is tight.

Caution

Do not tighten the knob on the MS front panel door after starting the vacuum system. Otherwise, it may be impossible to loosen the knob after the instrument is shut down.

Caution

When replacing a column with one of a larger internal diameter, a large volume of carrier gas may flow into the MS and prevent the vacuum system from starting. Before starting the vacuum system, adjust the pressure for the carrier gas and make sure that the flow into the column is less than 15 mL/min for Dual TMP model or less than 2 mL/min for Single TMP model, respectively.

(1) Click the **Vacuum Control** icon in the Assistant Bar. The "Vacuum Control" dialog box is displayed.

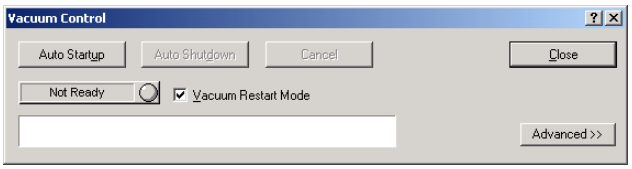


Figure 2.32 "Vacuum Control" Dialog Box

(2) Click the **Auto Startup** button.
The Startup indicator flashes, the progress bar is displayed, and the vacuum system startup sequence initiates. The various components start up in sequence, as noted in the progress bar, and when the vacuum system is ready for operation, "Completed." is displayed.

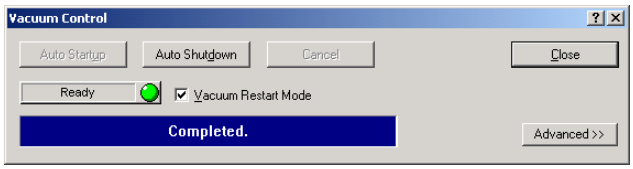


Figure 2.33 Vacuum System Startup Completed

(3) Close the "Vacuum Control" dialog box.

2.7 System Configuration

This section explains system configuration. Procedures are described for entering the properties for the autosampler, injection port, column, detector, additional temperature control, additional flow, and other installed components.

2.7.1 System Configuration

1. Click the Assistant Bar **System Configuration** icon to open the "System Configuration" dialog box.

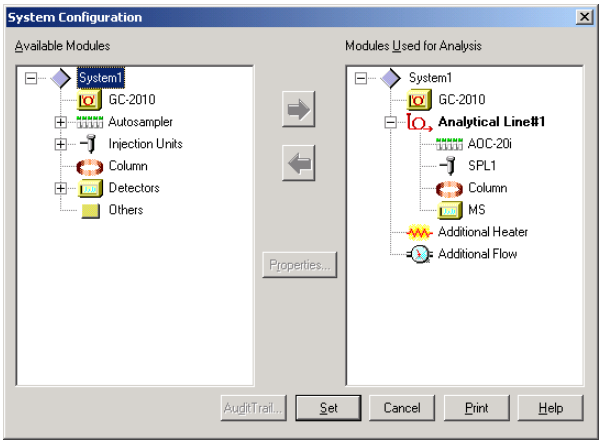


Figure 2.34 "System Configuration" Dialog Box

The components that can be included in the configuration are listed in the Available Modules box on the left. The tree structure includes the various GC, autosampler, injection port, detector, and additional options. To view the options that are available for a particular component, click the plus symbol (+) to the left of the respective icon.

2. To include a component in the configuration, select its icon in the Available Modules box and click the right arrow button. Alternatively, double-click the icon, or drag the icon from the left window and drop it in the right.
The current system configuration is displayed in the Modules Used for Analysis box on the right.

Available GCMS Modules

Module	Options
GC	GC-2010
Autosamplers	AOC-20i
	AOC-20i+s
	AOC-20d(M)
	AOC-20d(S)
Injection Ports	SPL
	OCI
	WBI
	PTV
Column	Column
Detectors	FID
	FPD
	FTD
	ECD
	TCD
	MS
Other Units	Other Units
Additional Heater*	
Additional Flow*	
* Units connected to the main unit are displayed automatically.	

3. Double-click each component listed in the Modules Used for Analysis box to open the "Properties" dialog box and enter the necessary parameters. Default settings may be used for most parameters. For column properties, selecting the type of column in use automatically enters its specifications. Ensure that the MS ion source is correctly selected. Change any other parameters according to the application and configuration. For more information about the parameters for each GC/MS system component, consult the online GCMS Help.

To print the configuration and properties, click the **Print** button in the "System Configuration" dialog box.

4. After all of the components included in the configuration are listed in the Modules Used for Analysis box and their properties are entered, click the **Set** button and exit the "System Configuration" dialog box.

2.8 System Check and Tuning

This section explains how to perform a system check and tune the GC/MS system. The system check is a diagnostic check that should be run before analysis to verify proper instrument operation. The system is tuned by selecting the appropriate items within the "Tuning" window.

2.8.1 System Check

Overview

Parameter	Description	Action if System Check fails
Maintenance	Verifies how long and how many times a GC/MS consumable part has been used.	Replace the part and reset its amount of use.
↓		
GC Check	Verifies system status of GC.	If adjustment or repairs are recommended, discuss them with your Shimadzu representative. If a part replacement is necessary, replace the part as recommended.
↓		
MS Check	Verifies various values related to MS analysis precision.	If adjustment or repairs are recommended, discuss them with your Shimadzu representative. If a part replacement or tuning is necessary, replace the part or tune as recommended.
↓		
Run Autotuning as Needed	Performs autotuning, when System Check fails.	
OR		
Run Autotuning	Performs autotuning regardless of System Check results.	
↓		
Report Out	Prints the System Check results.	

1. Click the Assistant Bar **System Check** icon to open the "System Check" dialog box.

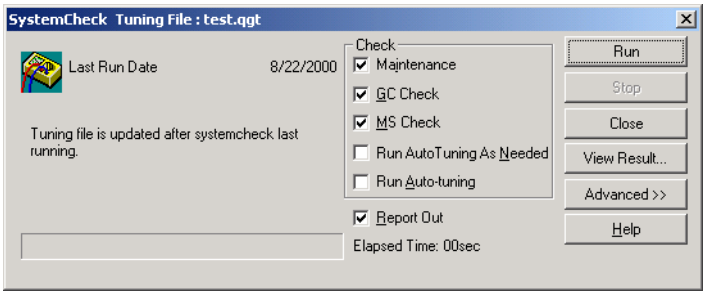


Figure 2.35 "System Check" Dialog Box

Note

The standards entered in System Check are the criteria used for the test, and do not reflect the manufacturer's specifications.
When autotuning is performed, the current tuning file is overwritten. Depending on the items being tuned, some previously obtained calibration curves can no longer be used.

"System Check" Dialog Box

The following items are displayed in the "System Check" dialog box.

Item	Description
Last Run Date	Displays the date on which the last system check was performed.
Result messages	Displays Pass, Fail and other messages.
Elapsed Time	Displays the time elapsed during the system check.

System Check

The system check is performed according to the items selected in the "System Check" dialog box.

Item	Description
Maintenance	Verifies how long and how many times a GC/MS consumable or maintenance part has been used.
GC Check	Verifies system status of GC.
MS Check	Verifies various values related to MS analysis precision.
Run Autotuning As Needed	If the "Signal intensity fluctuation," "Mass axis misalignment," or "Spectrum peak half-width fail" message is displayed, run autotuning.
Run Autotuning	Performs autotuning regardless of System Check results.
Report Out	Prints the System Check results.

To display the detailed settings for each item, click the **Advanced>>** button.

Note

Some checks may only be performed with an EI ion source. There are also some checks that may only be performed after the MS has completed operation. Checks that cannot be performed are disabled. For more information, please refer to GCMS Help.

Autotuning should usually be performed with the default parameters:

- "Resolution adjustment" selected
- "Spectrum peak half-width" = "0.6"
- "Sensitivity adjustment" selected
- "Mass Calibration" selected

Autotuning parameters may be changed by clicking the **Tuning** icon in the Assistant Bar. Select the Tuning file to be used in the advanced MS parameters tab.

The system is tuned according to the parameters entered in the Tuning file. Note that depending on the items being tuned, some previously obtained calibration curves can no longer be used.

Buttons

The following buttons are displayed in the "System Check" dialog box.

Button	Description
Run	Starts the system check.
Stop	Stops the system check. Only enabled while System Check is performed.
Close	Closes the "System Check" dialog box.
View result	Displays the results of the System Check. The results may be printed from the "System Check Result" dialog box.
Help	Opens the help file.
Advanced	Displays the advanced parameters. Only users authorized for "Changing System Check settings" can access these parameters.

The system check is initiated when Run is clicked. The time elapsed during the system check is displayed, and the progress bar tracks the system check progress.

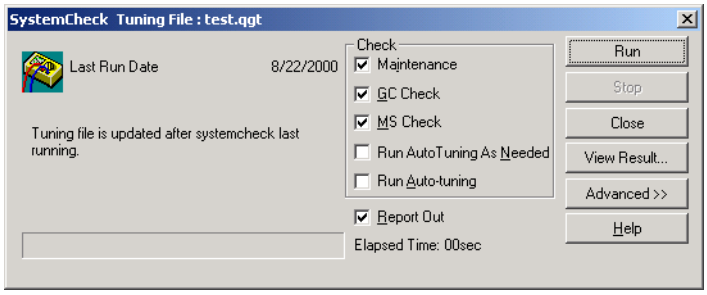


Figure 2.36 "System Check" Dialog Box

If the system check is completed without detecting any problems, "Pass" is displayed above the progress bar. If problems are detected, "Fail" is displayed.

2. Click the **Advanced>>** button to display the GC and MS advanced tabs. Select the GC tab to select the maintenance items and consumable parts to be checked during system check.

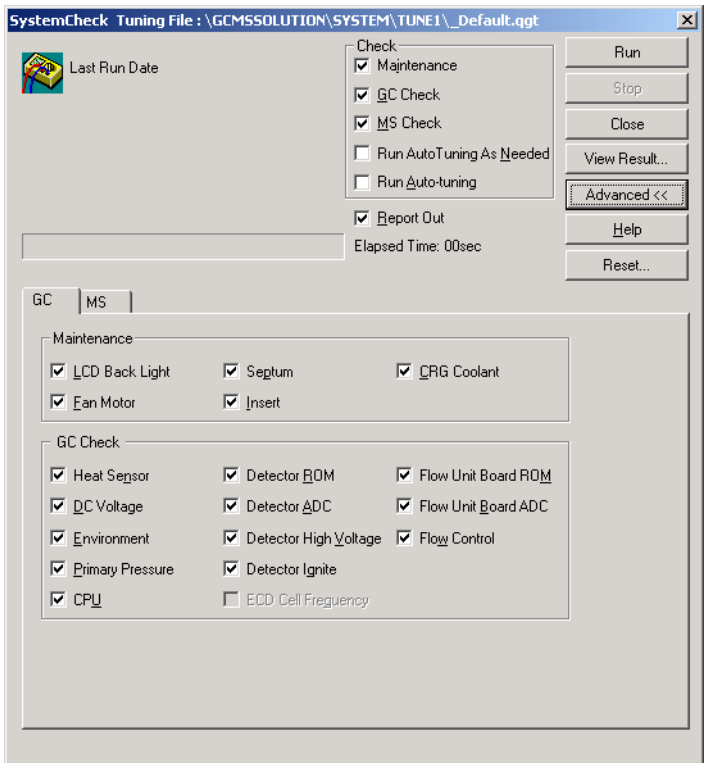


Figure 2.37 System Check GC Advanced Parameters Tab

GC Maintenance Items

Item	Description
LCD Back Light	Checks how long the back light has been active.
Fan Motor	Checks how long the fan motor has operated.
CRG Coolant	Checks the counter for CRG refrigerant depletion.
Septum	Checks how many times the currently installed septum has been used. For more information, refer to GCMS Help.
Insert	Checks how many times the currently installed glass insert has been used. For more information, refer to GCMS Help.

3. Select the MS tab to select the maintenance items and consumable parts to be checked during system check.

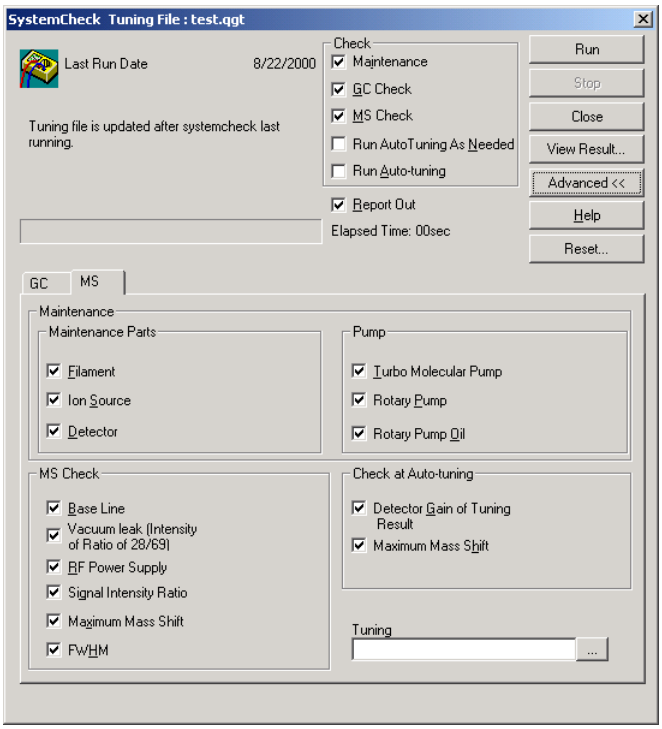


Figure 2.38 System Check MS Advanced Parameters Tab

MS Maintenance Items

Item	Description
Filament	Checks how long the filament has been used. For more information, refer to GCMS Help.
Ion Source	Checks how long the ion source has been used. For more information, refer to GCMS Help.
Detector	Checks how long the detector has been on and in use. For more information, refer to GCMS Help.
Turbo Molecular Pump	Checks how long the turbomolecular pump has been running and in use. For more information, refer to GCMS Help.
Rotary Pump	Checks how long the rotary pump has been running and in use. For more information, refer to GCMS Help.
Rotary Pump Oil	Checks how long the rotary pump oil has been used. For more information, refer to GCMS Help.
Reset	Opens the dialog box to set the use time or number of uses for maintenance parts.
	Note Only a Shimadzu representative can reset the turbomolecular pump use time.

The **Reset** button opens a dialog box to allow the use time or number of uses of a part to be reset. Click the **Clear** button to reset the length of time or number of times that a maintenance part has been used back to zero.

4. When the **Reset** button is clicked, the "Reset Consumables" dialog box is displayed. Reset the use time or number of uses any time a consumable part is replaced.

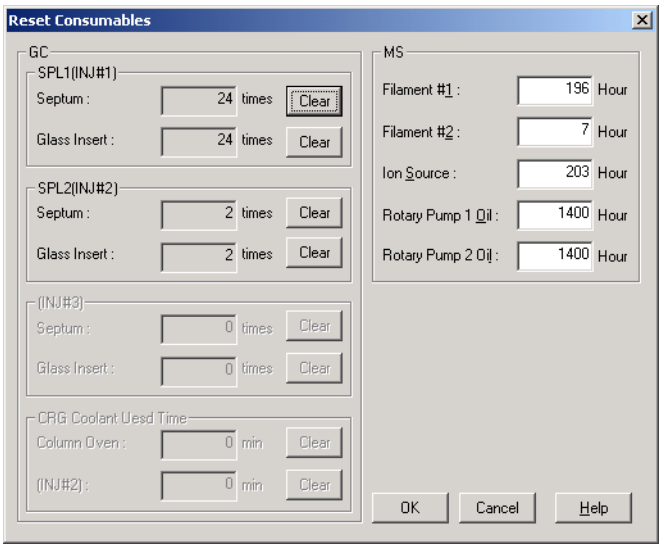


Figure 2.39 "Reset Consumables" Dialog Box

Note

Only users authorized for "Changing System Check Settings" can reset the time or number of uses.

- "0" is entered when the **Clear** button is clicked.
- If the part was changed earlier, enter the appropriate value.
- The value is changed when **OK** is clicked.
- The value displayed is the same value as when the "Reset Consumables" dialog box was last displayed. The values do not update automatically.

Reset Items

	Item	Description
GC	Septum	Reset the number of uses to 0 by clicking the **Clear** button. The number is not user definable.
	Glass Insert	
	CRG Refrigerant (Column Oven)	
	CRG Refrigerant (INJ#2)	
MS	Filament #1	Enter the appropriate time.
	Filament #2	
	Ion Source	
	Rotary pump 1 Oil	Enter the appropriate time.
	Rotary pump 2 Oil	This is only displayed if a second rotary pump is installed. Enter the appropriate time.

5. Click the GC tab and select the desired GC Check items.

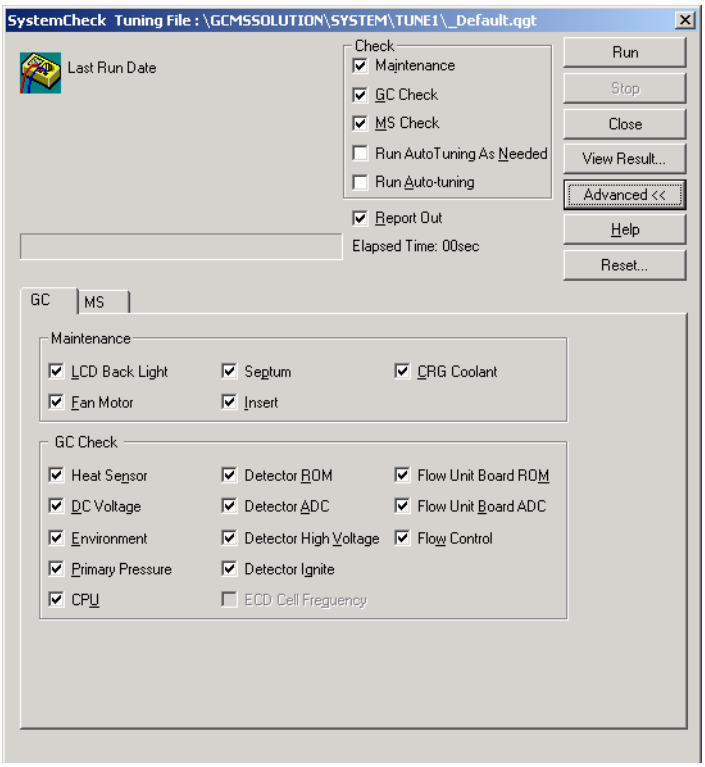


Figure 2.40 System Check Advanced GC Tab

GC Check Items

Item	Description
Heat Sensor	Checks how long the temperature sensor is used at 300 °C or above. Refer to GCMS Help for more information.
DC Voltage	Measures the DC voltage and checks if the error is within the standard range. Refer to GCMS Help for more information.
Environment	Measures the room temperature and air pressure, and checks if they are within the warranted operating conditions. Refer to GCMS Help for more information.
Primary Pressure	Measures the primary pressure of the carrier gas and checks if it is above the value of the environment setting. Refer to GCMS Help for more information.
CPU	Checks the CPU Register and Real-time Clock. Refer to GCMS Help for more information.
Detector ROM	Checks the detector ROM for each detector. Refer to GCMS Help for more information.
Detector ADC	Checks the detector ADC for each detector. Refer to GCMS Help for more information.

Item	Description
Detector High Voltage	Checks the high voltage of detectors respectively. Performed only when FID, FTD, or FPD is mounted. Refer to GCMS Help for more information.
Detector Ignite	Checks the Ignition Pulse and ignition procedure of each detector. Performed only when FID, FTD, or FPD is mounted. Refer to GCMS Help for more information.
ECD Cell Frequency	Measures the pulse voltage band for each detector ECD and compares dirtiness of cells with the threshold. Refer to GCMS Help for more information.
Flow Unit Board ROM	Checks ROM for each Flow Control Circuit (SLOT). Refer to GCMS Help for more information.
Flow Unit Board ADC	Checks ADC for each Flow Control Circuit (SLOT). Refer to GCMS Help for more information.
Flow Control	Checks if the flow is controlled properly for each Flow Port. Refer to GCMS Help for more information.

6. Click the MS tab and select the desired MS Check items.

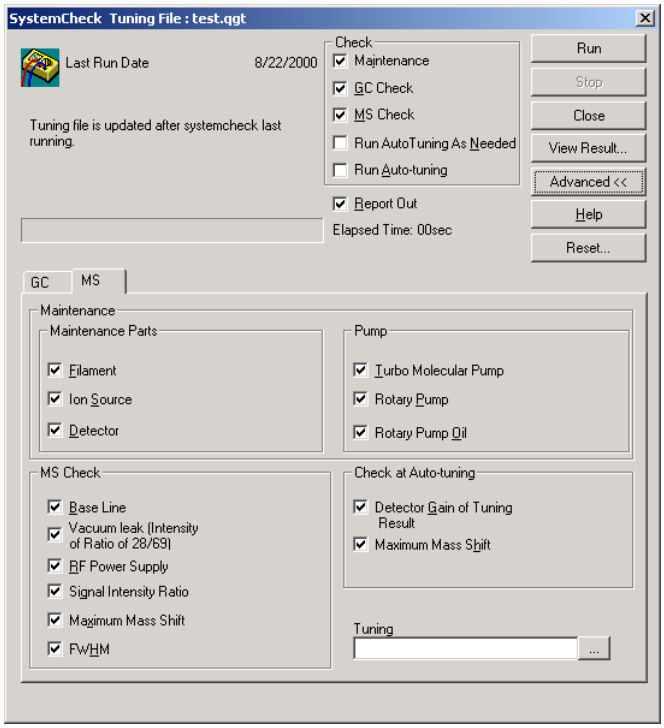


Figure 2.41 System Check MS Advanced Parameters Tab

Note

The parameters entered into the MS tab serve as the system check criteria, and do not reflect the manufacturer's specifications.

MS Check Items

Item	Description
Base Line	Determines the baseline fluctuation value (RMS). Refer to GCMS Help for more information.
Intensity Ratio of 28/69	Evaluates the ratio obtained from measuring m/z=28 (nitrogen) and m/z=69 (PFTBA). The ratio is used in determining if there is a leak. Refer to GCMS Help for more information.
RF Power Supply	Confirms that the high-frequency power is operating normally. Refer to GCMS Help for more information.
Signal Intensity Ratio	Compares the PFTBA signal intensity with the previous autotuning results. To apply the Adjust Sensitivity parameter entered in the Autotuning Conditions, "Mass Calibration" in the "Tuning Condition" dialog box must be selected. Refer to GCMS Help for more information.
Maximum Mass Shift	Determines if the mass axis has shifted. For this parameter to be corrected during autotuning, "Calibrate Mass" must be selected in the Autotuning Conditions. Refer to GCMS Help for more information.
FWHM	Determines the spectrum peak width at half-height shift from the mass peak width at half-height values entered for m/z = 69, 219, and 512. For this parameter to be corrected during autotuning, "Adjust Resolution" must be selected in the Autotuning Conditions. Refer to GCMS Help for more information.

7. Select the desired items to be checked during autotuning in the "Check at Autotuning" section of the MS tab.

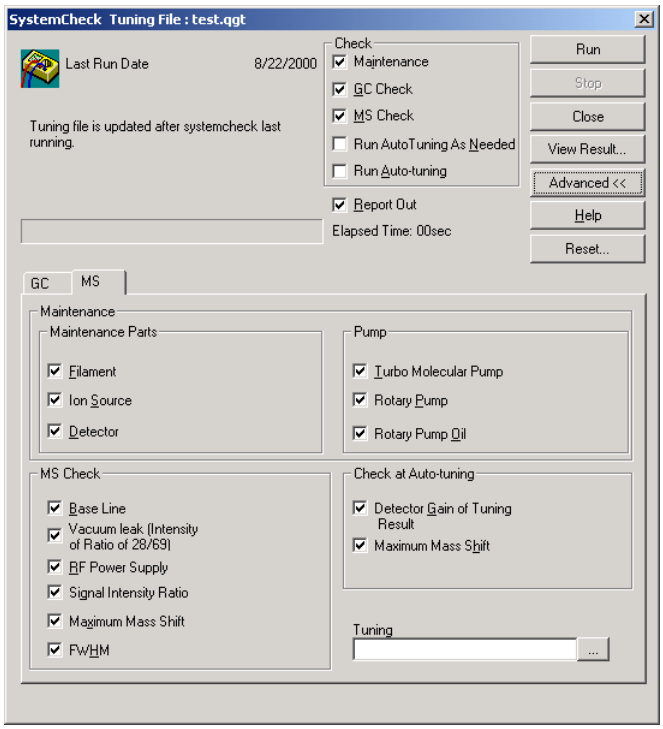


Figure 2.42 System Check MS Advanced Parameters Tab

When "Run Autotuning as Needed" or "Run Autotuning" are selected, these items are checked after autotuning is performed.

Checks after Autotuning

Item	Description
Detector Gain of Tuning Result	Determines the detector voltage setting that resulted from autotuning. Refer to GCMS Help for more information.
Maximum Mass Shift	Determines the mass axis shift. Note that "Calibrate Mass" must be selected in the Autotuning Conditions. Refer to GCMS Help for more information.

8. Select the tuning file to use for autotuning during the system check.

Tuning File

Item	Description
Tuning	The file displayed in Tuning is used to autotune the instrument during the system check. To select or change the file, either enter the full file path directly or open the "Select Tuning File" dialog box by clicking the browse button to the right. From the "Select Tuning File" dialog box, select the appropriate file and click **OK**. The file name is now displayed in Tuning.

9. Click the **Run** button to run the system check.

10. Select **View Result...** to display the "System Check Result" dialog box. This displays the results of the last system check.

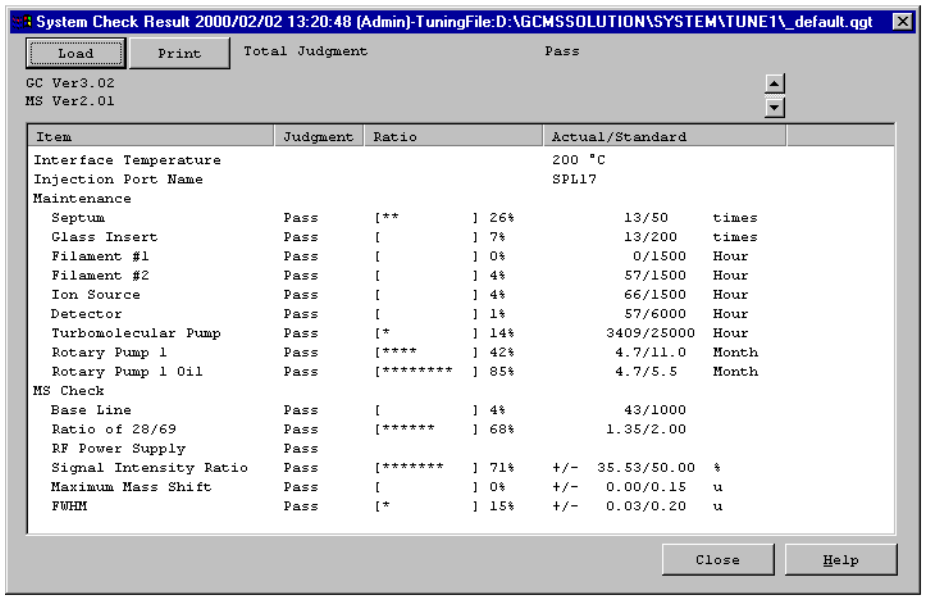


Figure 2.43 "System Check Result" Dialog Box

Note

After performing a check, the results are displayed automatically. Current and previous system check results can be printed. It is not necessary to run the system check to display or print previous results.

System Check Table

Columns	Descriptions
Item	The check item that was performed.
Judge	Whether the item passed or failed the test, and suggested actions.
Ratio	The ratio of the measured value to the standard value. Allows quick examination of system condition.
Actual/Standard	Measured and standard values for the item.

Buttons in the "System Check Result" Dialog Box

Button	Description
Load	Open past results that were saved in the log folder. The files are saved and named automatically. A 14-character name is assigned. The first character is the system number. The second is an underscore (_). The third through sixth are the year. The seventh and eighth are the month. The ninth and tenth are the day. Characters 11 and 12 are the hour time, and 13 and 14 are the minutes. Thus, if a system check is performed on System 1 on January 1, 2000, at 13:00, the name is 1_200001011300.qgc.
Print	Print the currently displayed results.

11. Click the **Print** button to print the currently displayed system check results.
The maintenance, GS, MS and post-tuning check items and their results are printed, followed by specific GC and MS information.

Text Printed with System Check Results

Text	Description
Header	Prints the user name, date and time of system check, and time of printing.
Footer	Prints the page number and number of pages in the center.

2.8.2 Tuning

In the "Tuning" window, the parameters controlling the MS can be adjusted so that the desired mass spectrum peaks are obtained.

There are two tuning methods: autotuning and manual tuning. Autotuning automatically adjusts the various instrument control parameters as specified in the Autotuning Conditions. In manual tuning, various control parameters are used to manually adjust the mass peaks. When the "Tuning" window is opened, the tuning file that was last saved or used for data acquisition is displayed. Unless otherwise specified, the file that opens automatically in the "Tuning" window is used during system checks or data acquisition.

Operations associated with creating or accessing a tuning file are described below.

1. Click the Assistant Bar **Tuning** icon in the "GCMS Real Time Analysis" window. The "Tuning" window is displayed.

   To create a new tuning file, select New Tuning File from the File menu. Proceed to Step 2.

   To perform tuning with an existing file, select **File > Open Tuning File** to display the "Open Tuning File" dialog box. Select the appropriate tuning file, and click **Open**. Proceed to Step 3.

2. Click the Assistant Bar **Autotuning Condition** icon, or select **Tuning > Condition of Autotuning**, to open the "Tuning Information" dialog box.

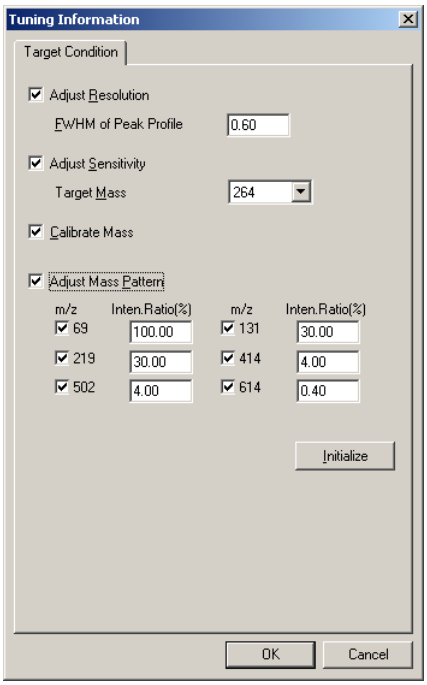


Figure 2.44 "Tuning Information" Dialog Box

Tuning Condition

Select which parameters to apply during autotuning, and enter those parameters. The parameters include Adjust Resolution, Adjust Sensitivity, Calibrate Mass, and Adjust Mass Pattern. Select the parameters to include during autotuning. When the tuning conditions are changed, the MS system check or autotuning results may vary.

Instrument Parameter	Description
Adjust Resolution	Adjust the resolution so that the peak width at half-height value obtained with the standard (PFTBA) is near the value entered for Spectrum peak width at half-height (FWHM).
FWHM of Peak Profile	0.3 - 2.0 u (where "u" denotes atomic mass unit) Note: Although resolution increases as the width at half-height value decreases, the signal intensity tends to decrease.
Adjust Sensitivity	Adjust the lens system to the maximum sensitivity for a specific ion with the Target Ion adjustment.
Calibrate Mass	A spectrum produced from a standard (PFTBA) is used to correct for mass axis misalignment.
Adjust Mass Pattern	Adjust the lens system to maximize the m/z 502 peak, and correct the intensity to equalize the intensity pattern between the spectrum of the standard measured by MS in the magnetic field type and the spectrum in the main instrument. Pattern calibration sets the intensity correction parameter to correspond to the specified intensity ratio.

After selecting the appropriate parameters and entering values as necessary, click the **OK** button and close the "Tuning Information" dialog box.

3. Click the Assistant Bar **Start Autotuning** icon or the **Start Tuning** button on the toolbar to tune the instrument automatically. Autotuning can also be started with the **Tuning > Start Autotuning** command.

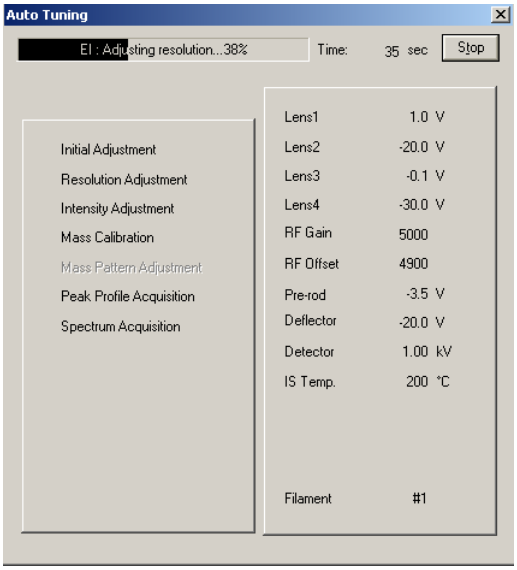


Figure 2.45 "Autotuning" Dialog Box

After autotuning is completed, save the file. Select **File > Save Tuning File As** the first time the file is saved or to save the file with a new name. Select **File > Save Tuning File** to overwrite the existing tuning file.

4. Click the Assistant Bar **Tuning Result View** icon to view the tuning results. The peak profile corresponding to each m/z is displayed.

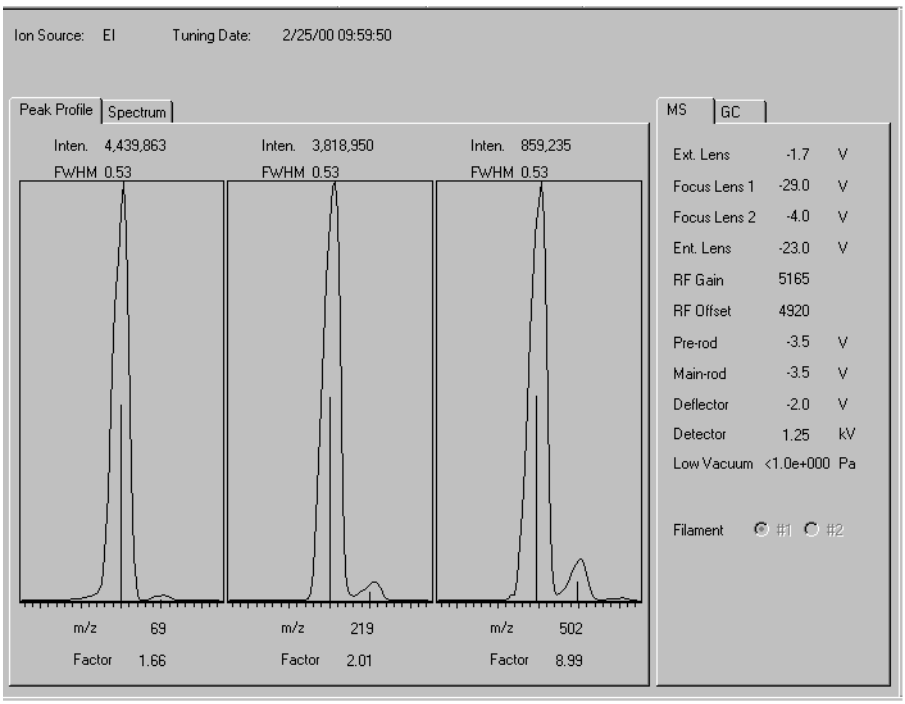


Figure 2.46 Tuning Results - Peak Profiles

5. Click the button in the toolbar to print the tuning results.

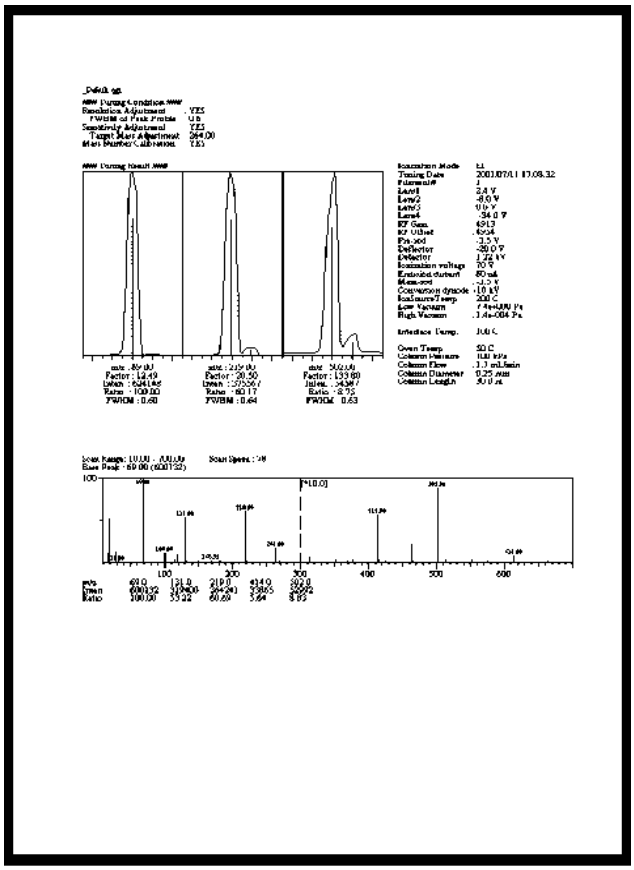


Figure 2.47 Printed Tuning Report

Note

The "Peak Monitor" screen displayed by clicking the Assistant Bar **Peak Monitor View** icon is the same as the screen displayed during the Vacuum Leak Check and other operations.

Peak Monitor View

For more information about checking for vacuum leaks, refer to Section 10.6 "Checking for Leaks", page 228.

2.8.3 Adjustment for High Masses

This instrument incorporates an auto-tuning function that conducts mass calibration for molecular weight up to m/z 614. Mass displacement is rare in the normal range of operation (up to approximately m/z 700.)
However, displacement from the calibration for the m/z is possible in the high mass range above m/z 700.
To ensure accurate calibration in the range above m/z 700 requires manual calibration by the following procedure.

Analysis Conditions (Column: DB-5 ms, Inner diameter 0.25 mm, Length 30 m, Thickness 0.25 µm)

Sample : Tris (perfluoroheptyl)-S-triazine 1 % solution (acetone)
GC : INJ 250 °C, Column head pressure 100 kPa, Total flow rate 30 mL/min, Splitless injection (1 min. sampling)
COL 50 °C (1 min) _ 5 °C /min_100 °C (1 min)_30 °C /min_250 °C (10 min)
MS : I/F 250 °C , Ion source 200 °C

1. Execute the autotuning command.
After it has been completed, open the Peak Monitor window, and turn the filament off.

2. Inject the 1 µL triazine sample, and start the GC.

3. In the mass calibration window opened from the Peak Monitor window, align the cursor with the highest cell for m/z 866.

4. When 8 minutes have elapsed since the sample injection, turn the filament on.

5. After 10 minutes have elapsed, a 3-minute segment of peaks can be calibrated. Click the **Centering** button to calibrate the peak.

6. Click the **Supplement** button, then click **OK**.

7. Save the tuning file. For the message of "Do you acquire and save peak profile/ spectrum data?", click the **No** button.

Note

Traces of the injected triazine may remain in the INJ, column, etc. and hinder subsequent analysis after calibration. In that case, set the column temperature to 250 °C and proceed with aging. Although a sample of about 1 ppm can be analyzed for about 1 hour by aging, changing the insert or aging for several hours are recommended to conduct ultra-microanalysis.

2.9 Common Operations

2.9.1 Managing Files

1. File types

Type	Description	Ext.
Data file	Saves raw acquired data, such as chromatograms and spectra, including: • Results of peak integration and concentration calculations • The oven temperature status and errors generated during data acquisition • The contents of the method file used in analysis, including parameter settings • The contents of the report format file when reports are generated • The contents of the batch file when batch processing is performed • The contents of the tuning file used in analysis	.qgd
Method file	Saves parameters related to the instrument, data acquisition, qualitative, quantitative, data view, and QA/QC. System configuration is saved so that it can be checked when the method file is opened. Calibration curves used in the method are also saved.	.qgm
Report format file	Saves which information to include in the report and how to lay out the information, along with other report format information such as headers and footers.	.qgr
Batch file	Saves the batch tables used for automating data acquisition and analysis. GCMS Real Time Analysis and GCMS Postrun Analysis can access the same file.	.qgb
Tuning file	Saves the tuning parameters, used to adjust the instrument, and the results of tuning using these parameters.	.qgt
Library file	Saves spectra and compound information for similarity searches. Library files include the optional NIST commercial database and private libraries created from the spectra and compound information of target compounds. The spectra and information in library files is searched for matches with a spectrum.	.lib*1
Browsing file	Saves the layout information such as loaded method/data file names, order of files, and presence of statistic calculation settings.	*.qgq
Layout file	Saves the layout, the data loaded to each cell, and the display-related information.	*.lyt

*1 The library is actually composed of multiple files. The constitutive files are somewhat different for commercial and private libraries. When copying library files, ensure that all associated files are copied.
Commercial library: .lib, .c2s, .fom, .nam, .ncv, .spc, .str
Private library: .lib, .com, .flg, .fom, .nam, .spc

2. File management

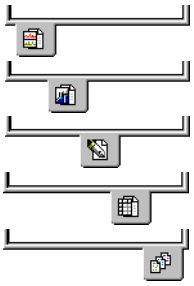
The "Data Explorer" window displays the various files by type for easier management. The five tabs of the Data Explorer are Data, Method, Report Format, Batch, and All Files. Any file can be moved, copied or deleted from the "Data Explorer" window without returning to the Windows desktop.

(1) Opening the "Data Explorer" Window

Select **File > Data Explorer** to open the "Data Explorer" window. When the Data Explorer is displayed, a check appears to the left of Data Explorer in the File menu to show that it is visible. Selecting **File > Data Explorer** when it is checked closes the "Data Explorer" window.

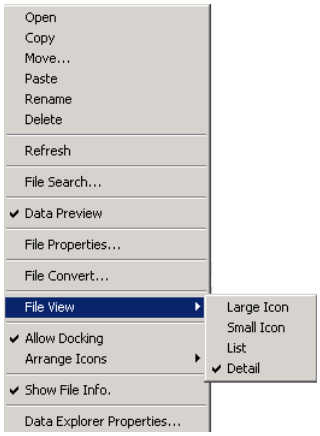
(2) Displaying a File Type

Click the tab that corresponds to the appropriate file type. The tabs, located beneath the Data Explorer list box, are Data, Method, Report Format, Batch, and All Files. The files of the selected type are displayed.



(3) Changing how Files are Displayed

Change the way files are displayed by right-clicking the list box and selecting **File View**. From the File View submenu, select how files are displayed from **Large Icon**, **Small Icon**, **List**, and **Detail**. The way files are displayed can also be changed with the **View > File View** command when the "Data Explorer" window is active.

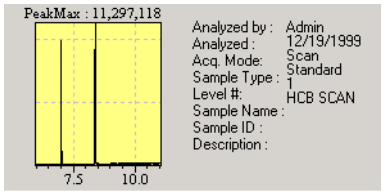


Note

The check mark to the left of the File View submenu indicates the current display mode.

(4) Confirm the data.

The data preview is displayed below the list box by right-clicking the list box and selecting **Data Preview**. Chromatogram and sample information are displayed when the Data Tab is selected. File comments are displayed if other tabs are selected.



(5) Changing the Project

To change the directory in which the files are located, or the project, click the **Select Project** button to the far right of the Project in combo box. The "Project (Folder) Selection" dialog box is displayed.

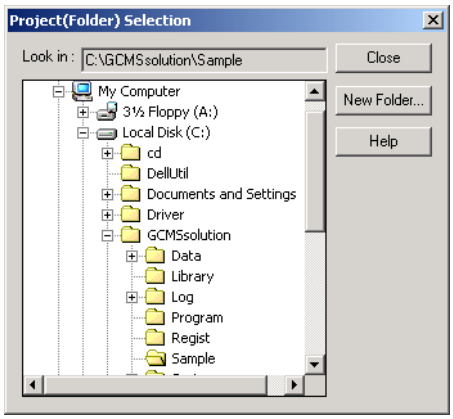


Figure 2.48 "Project (Folder) Selection" Dialog Box

When the "Project (Folder) Selection" dialog box is displayed, the open folder is the project currently selected. The levels are arranged as in Windows Explorer. Select the desired folder, and click the **OK** button. The "Project (Folder) Selection" dialog box is closed, and the files in the newly selected project are displayed in the list box.

(6) Creating New Projects

Click the **Create New Project** button, located to the left of **Select Project** button, to open the "Create New Project (Folder)" dialog box.

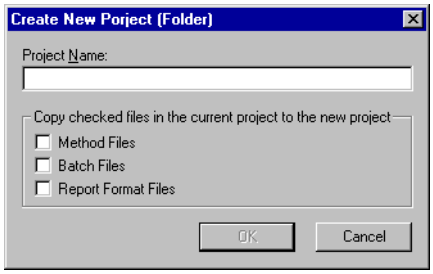


Figure 2.49 "Create New Project (Folder)" Dialog Box

Enter a name for the project. To copy method, batch and/or report format files from the project that is currently open, select the appropriate check boxes.
After entering the name and selecting the files to copy, click the **OK** button. The "Create New Project (Folder)" dialog box is closed, and the files that were copied (if any) are displayed in the list box.

2.9.2 Opening and Saving Files

This section describes how to open and save files, such as data or method files. The Open and Save commands are similar to those used in Windows. Refer to the Windows manual for more information.

1. Opening a File

Although the procedure below describes opening a method file, it applies to all files.

(1) Select **File > Open** or **File > Open Method File**, or click the toolbar **Open** button. The "Open Method File" dialog box is displayed.

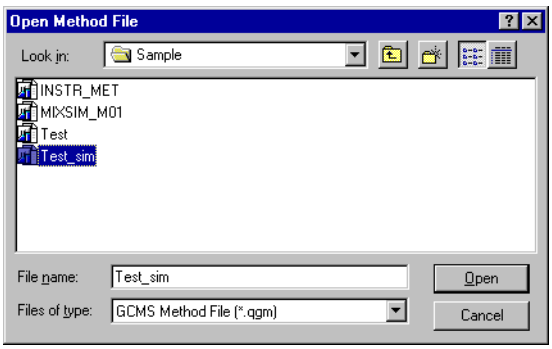
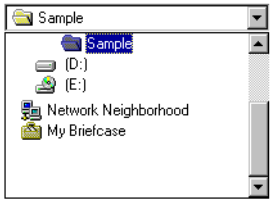


Figure 2.50 "Open Method" File Dialog Box

(2) Click the "Look in" combo box button, and scroll up and down to change directories or drives. Click the **Up One Level** button to open the directory containing the currently opened folder. To open a lower directory, select a folder from the list box and click the **Open** button, or double-click the folder. Change and expand directories until the folder in which the desired file has been saved is displayed in the combo box.



(3) Select the desired file from the list box, and confirm that the correct file name is displayed in the File Name text box. Click the **Open** button to open the selected file.

2. Saving a New File

Although the procedure below describes saving a newly created data file, it applies to all files.

(1) Select **File > Save Data File As** to open the "Save Data File As" dialog box.

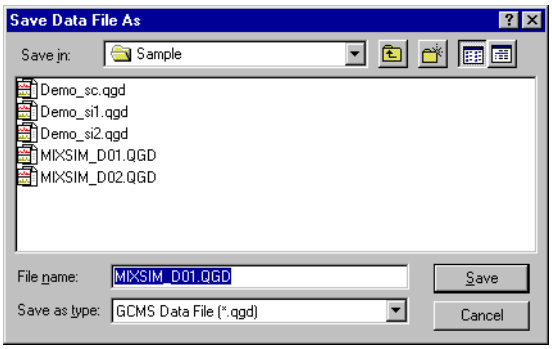
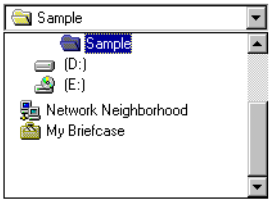


Figure 2.51 "Save Data File As" Dialog Box

(2) Click the "Save in" combo box button, and scroll up and down to change directories or drives. Click the **Up One Level** button to open the directory containing the currently opened folder. To open a lower directory, select a folder from the list box and click the **Open** button, or double-click the folder. Change and expand directories until the folder in which the desired file has been saved is displayed in the Save in combo box. If necessary, create a new folder by clicking the **Create New Folder** button.



(3) Enter a file name in the File Name text box, and click the **Save** button to save the file using that name.

2.9.3 Using Help

1. Opening help files to a specific topic

To access information about the active window or dialog box, press the F1 key on the keyboard. If available, the GCMS Help window will open to the topic that corresponds to the active window or dialog box.

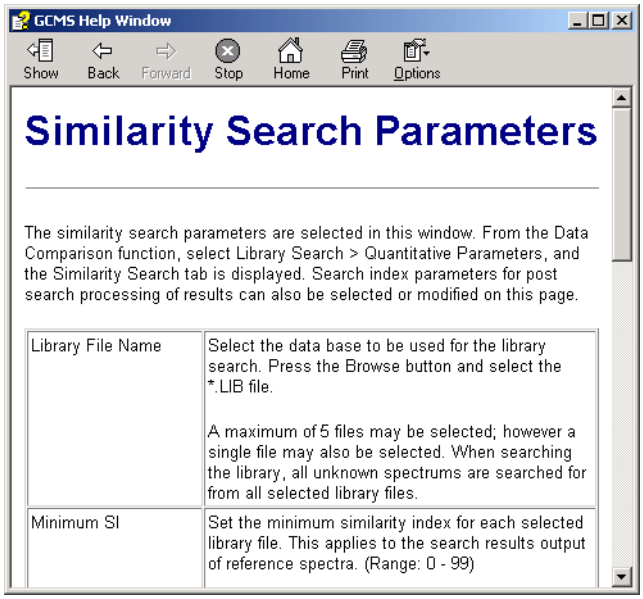


Figure 2.52 GCMS Help Window

Additionally, some windows and dialog boxes include a help button that, when clicked, links directly to the appropriate help topic.

2. Opening help files by keyword
Select **Help > Contents** to open the GCMS Help window.

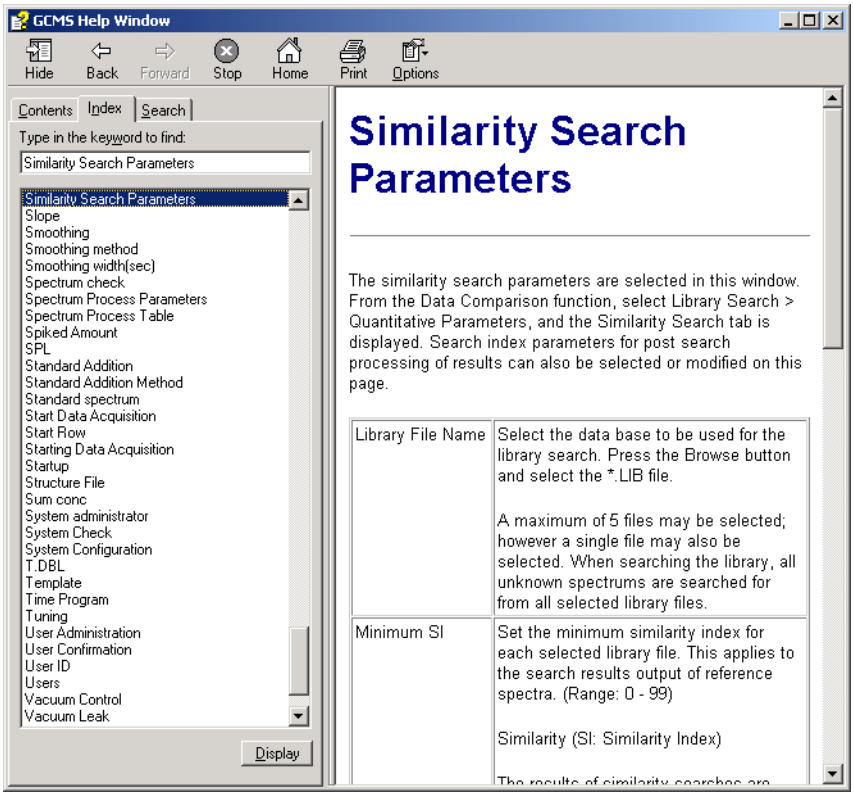


Figure 2.53 GCMS Help Contents Window

Click the Index tab to the left of GCMS Help window. An index of keywords is displayed in the list box.
Scroll the list box to search for the desired topic. Select the keyword, and click the **Display** button to open the help topic.

Note

When the same keyword appears in more than one topic, the list of Topics Found is displayed. The list is displayed in the right side of the GCMS Help window. Scroll to search for the desired topic. Select the topic from the list, and click the **Display** button.

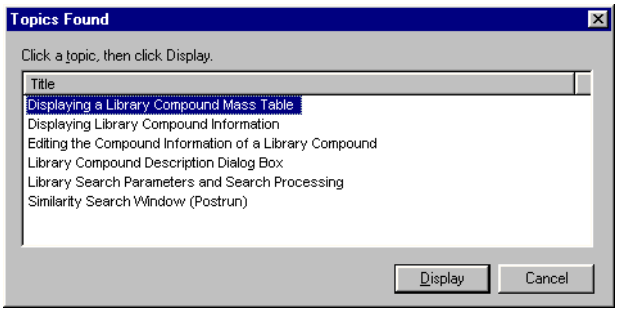


Figure 2.54 List of Topics Found

Note

The GCMS Help window can be moved and resized as desired. To change locations, drag the window using the title bar. To change the size, either drag the frame or square corner on the lower right of the window.

2.9.4 Concepts of System Administration

In the GCMS-QP2010, system security revolves around the idea of user management. The GCMSsolution software allows an administrator to select an authorization level for each user. The authorization level determines the amount of access the user has to the instrument, data and methods. This prevents users who should not change methods from inadvertently doing so, and protects against the system being shut down by a user who does not have the authority to make such a decision.

1. Users and Groups

System administration involves users, groups, and an authorization system. The diagram below illustrates the user, group and authorization system. Each user belongs to one or more groups. Each group is assigned certain levels of authorization. Each authorization level is composed of a defined set of assigned rights.

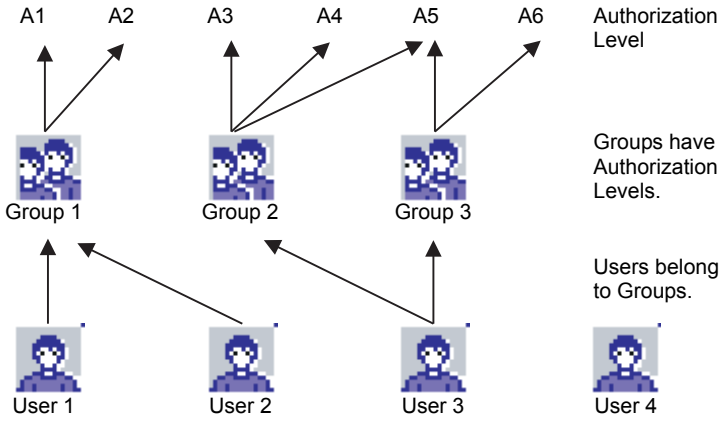


Figure 2.55 Users, Groups and Authorization Levels

- User 1 belongs only to Group 1, and can operate under Authorization 1 and Authorization 2 held by Group 1.
- Like User 1, User 2 belongs only to group 1, and can also operate under Authorizations 1 and 2.
- User 3 belongs to Group 2 and Group 3, and can operate under Authorizations 3 - 6, which are all of the authorizations held by the two groups.
- User 4 does not belong to any group, and thus does not have any authority.

Authorization Level	Description	Default groups		
		H/W Administrator	Method Developer	Operator
User Administration	Authority to add or delete users, set policy, and perform functions described in Section 2.9.5 "System Administration", page 71.			
Edit System Configuration	Authority to change system configuration and perform functions described in Section 2.7 "System Configuration", page 39.	×	×	
Edit Method (Instrument Parameter)	Authority to edit the instrument parameters entered in the method file, and perform functions described in Chapter 2 "Basic Operation" on page 11.		×	
Edit Method (Acquisition Display Settings)	Authority to edit the Data View Parameters in the method file, or the parameters accessed with the **View > Data View Parameters**.		×	
Edit Method (Data Processing Parameters)	Authority to edit the Qualitative and Quantitative analysis parameters in the method file, or the parameters described in Chapter 4 "Qualitative Analysis" on page 111 and Chapter 5 "Quantitative Analysis" on page 137.		×	
Edit Report Format	Authority to edit report format files and perform functions described in Chapter 6 "Generating Custom Reports" on page 165.		×	
Edit Batch Table	Authority to edit batch tables and perform functions described in Chapter 7 "Continuous Analysis" on page 175.		×	×
Create Template	Authority to create templates for method, batch and report format files, and perform functions described in Section 2.9.6 "Templates", page 81.		×	
Modify Autotuning Settings	Authority to change tuning conditions and perform functions described in Section 2.8.2 "Tuning", page 54.	×		
Modify System Check Settings	Authority to change system check parameters and perform functions described in Section 2.8.1 "System Check", page 41.	×		
ON OFF Vacuum System	Authority to start up and shut down instruments, and perform functions described in Section 2.4 "Instrument Startup and Shutdown", page 14.	×		
Run System Check and Autotuning	Authority to perform system checks and autotuning as described in Section 2.8 "System Check and Tuning", page 41.	×	×	×
Run Peak Monitor	Authority to perform peak monitoring.	×		

Authorization Level	Description	Default groups		
		H/W Administrator	Method Developer	Operator
Run Batch Data Acquisition	Authority to perform automated data acquisition and analysis as described in Section 7.2 "Automated Data Acquisition and Data Analysis", page 177.		×	×
Run Single Data Acquisition	Authority to acquire data for a single sample and perform functions described in Section 3.3 "Single Run Setup", page 104.		×	
Run Postrun Analysis	Authority to analyze data in GCMS Postrun Analysis and perform functions described in Chapter 4 "Qualitative Analysis" on page 111 and Chapter 5 "Quantitative Analysis" on page 137.		×	×

2. Administrators
Administrators are users that have full authority to perform all functions included in the GCMSsolution software. When the software is installed, a default user with the user ID "Admin" and Administrator privileges enables the system administrator to access and setup the software.

3. Using Shimadzu User Authentication Tool
If you use the CLASS-Agent software (optional), you can manage user information using Shimadzu User Authentication Tool. Shimadzu User Authentication Tool allows you to share the information of users with other software also using Shimadzu User Authentication Tools, such as CLASS-Agent and GCsolution. To utilize Shimadzu User Authentication Tool, set "Shimadzu User Authentication Tool" in the "User Authentication Mode Select" window at the installation of the GCMSsolution. For details of the installation and environmental setting of Shimadzu User Authentication Tool, refer to the instruction manual that comes with Shimadzu User Authentication Tool.

Note

To manage user information using the Shimadzu User Authentication Tool, be sure to install the Shimadzu User Authentication Tool in Version 1.08 or later versions.

Note

If you select to use the Shimadzu User Authentication Tool when the GCMSsolution is already in actual operation, current user information will be replaced with that registered in the Shimadzu User Authentication Tool. Note therefore that if users are registered in the GCMSsolution but not in the User Shimadzu Authentication Tool, it is necessary to newly register them to the Shimadzu User Authentication Tool. By referring to Section "2.9.5 System Administration", "Users" dialog box, reregister the users. Note too that if user IDs or user names are identical between the User Databases of the GCMSsolution and the Shimadzu User Authentication Tool, the user information (e.g. password) specified with the Shimadzu User Authentication Tool becomes effective. Be sure to review the relevant user information by referring to Section "2.9.5 System Administration".

2.9.5 System Administration

The "System Administration" dialog box enables the administrator to perform a number of functions. New users are added and their user names, passwords, and access rights assigned. Groups are added or edited. Parameters controlling user log-in are set. Whenever changes are made or users added, an event log records the modification.

1. Select **File > User Administration** to open the "System Administration" dialog box.

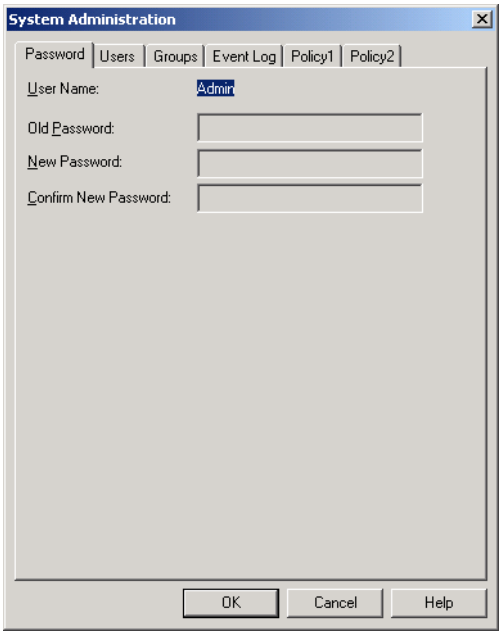


Figure 2.56 "System Administration" Dialog Box

2. The Password tab allows the password for a user registered in the system to be changed. If a user does not have a password assigned, all of the fields on the tab are disabled.

Parameter	Description
User Name	Displays the name of the user that is currently logged in.
Old Password	Enter the password that is currently in use.
New Password	Enter a new password.
Confirm New Password	Enter the new password exactly as it was entered in New Password to confirm that it was entered correctly.

Note

If a user logs in without a password, the User tab is used to enter a password. For more information, see the next paragraph, which describes the User tab.

3. The User tab displays a list of all users that have been registered in the system. Select a user name to display the groups to which the user belongs in the Assigned Groups box. To add a user, click the **Add** button. The "Add User" dialog box is displayed.

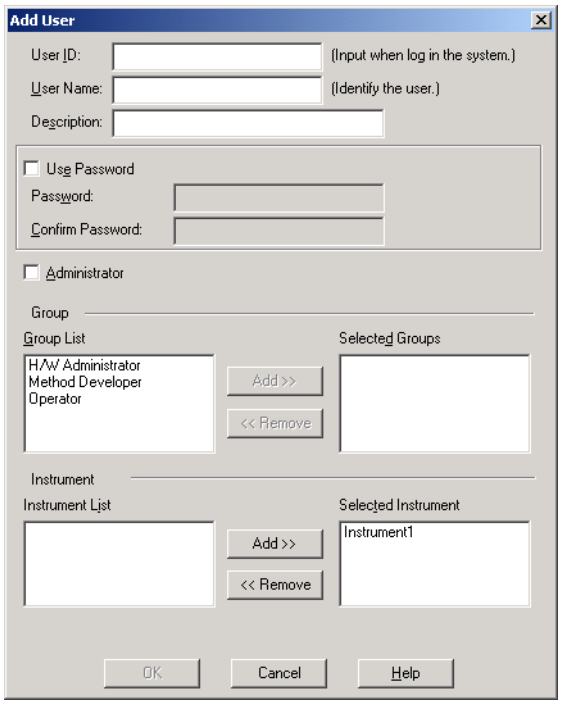


Figure 2.57 "Add User" Dialog Box

Enter the following information for each user.

Property	Description
User ID	Type the user ID for the new user. This is used for login.
User Name	Enter a user name for the new user.
Description	Enter the descriptions of the user, e.g. full name.
Use Password	Check here to have the user enter a password at login. When checked, the Password and Confirm Password fields are enabled.
Password	If Use Password is checked, enter a password for the user to enter at login.
Confirm Password	Enter the password a second time to confirm that it was entered correctly.
Administrator	Check here to make the user an administrator.
Group List	Select the group(s) to which the user will belong and click the **Add** button, or double-click the group. The group is moved to the Selected Groups box.
Selected Groups	Displays the groups to which the user belongs. To remove the user from a group, select the group and click the **Remove** button, or double-click the group.

Note

Set the minimum length for a password in the Policy 1 tab, if passwords are used.

Click the **OK** button after setting up the new user, and the "Add User" dialog box is closed.

To delete a user, select the appropriate user in the Users tab and click the **Remove** button.

To display the properties for a user, select the appropriate user in the Users tab and click the **Properties** button. The "User Property" dialog box is displayed.

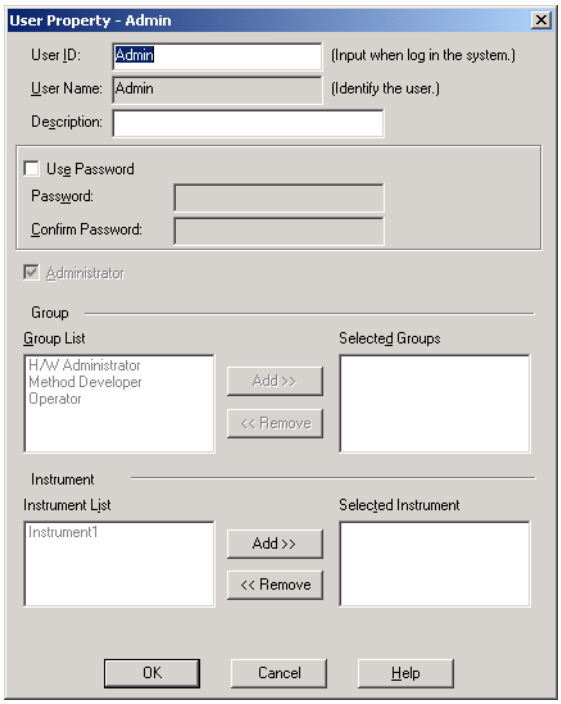


Figure 2.58 "User Property" Dialog Box

The following properties are displayed for each user.

Property	Description
User ID	Displays the user ID of the selected user. This cannot be changed.
User Name	Displays the user name of the selected user.
Description	Displays the descriptions of the selected user.
Use Password	Check here to have the user enter a password at login. When checked, the Password and Confirm Password fields are enabled. If it was already checked, the entered password is currently in use.
Password	If Use Password is checked, enter a password for the user to enter at login.
Confirm Password	Enter the password a second time to confirm that it was entered correctly.
Administrator	Check here to make the user an administrator. If it was already checked, the user currently has Administrator privileges.
Group List	Select the group(s) to which the user will belong and click the **Add** button, or double-click the group. The group is moved to the Selected Groups box.
Selected Groups	Displays the groups to which the user belongs. To remove the user from a group, select the group and click the **Remove** button, or double-click the group.

Note

Set the minimum length for a password in the Policy 1 tab, if passwords are used. Click the **OK** button after editing the user properties, and the "User Property" dialog box is closed.

4. The Groups tab displays a list of all entered groups. Select a group to display the rights authorized for that group. To create a new group, click the **Add** button. The "Add Group" dialog box is displayed.

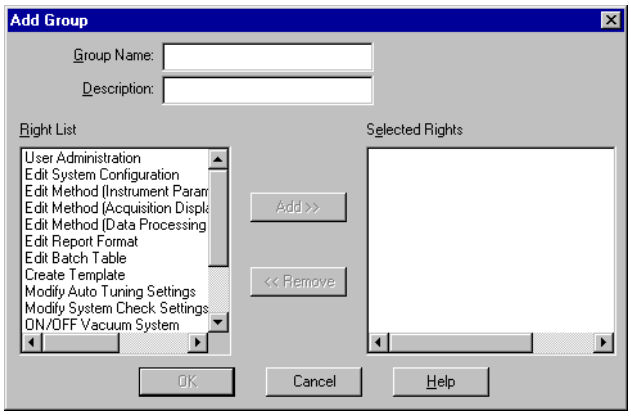


Figure 2.59 "Add Group" Dialog Box

Enter the following information for each new group.

Property	Description
Group Name	Enter a name for the new group.
Description	Enter a brief description of the new group.
Right List	Select the Right(s) to which the group will have authorization and click the **Add** button, or double-click the Right. The Right is moved to the Selected Rights box.
Selected Rights	Displays the Rights to which the group has authorization. To remove a Right, select it and click the **Remove** button, or double-click the Right.

Click the **OK** button after setting up the group, and the "Add Group" dialog box is closed.

To delete a group, select the appropriate group in the Groups tab and click the **Remove** button.

To display the properties for a group, select the appropriate group in the Groups tab and click the **Properties** button. The "Group Properties" dialog box is displayed.

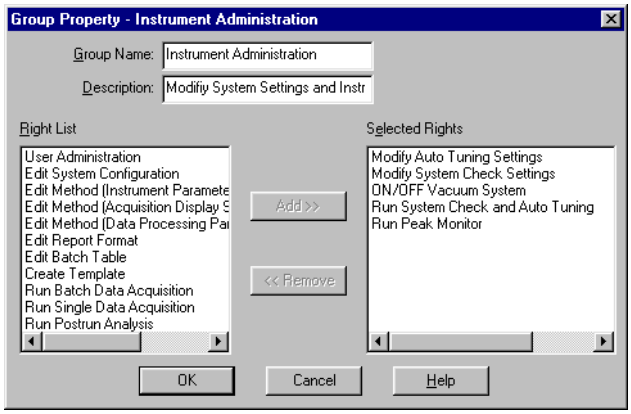


Figure 2.60 "Group Properties" Dialog Box

The following properties are displayed for each group.

Property	Description
Group Name	Displays the name of the selected group.
Description	Displays a brief description of the selected group.
Right List	Select the Right(s) to which the group will have authorization and click the **Add** button, or double-click the Right. The Right is moved to the Selected Rights box.
Selected Rights	Displays the Rights to which the group has authorization. To remove a Right, select it and click the **Remove** button, or double-click the Right.

Click the **OK** button after editing the group properties, and the "Group Property" dialog box is closed.

5. The Event Log tab displays the history of all changes and actions made in User Administration. To use the log with other applications or GCMSsolution function, save it as a text file by clicking the **Save As Text** button. The "Convert Event Log" dialog box is displayed.

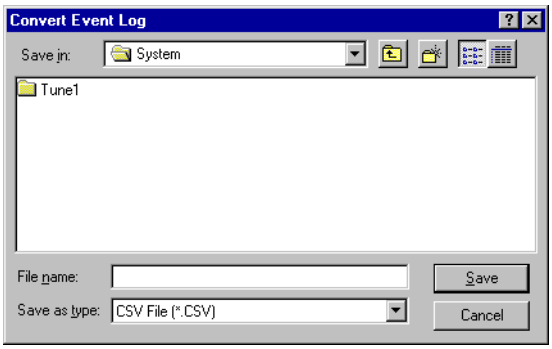


Figure 2.61 "Convert Event Log" Dialog Box

Select a directory, enter a file name, and click the **Save** button to save the file. Refer to Section 2.9.2 "Opening and Saving Files", page 63 for more information.

To delete the Event Log, click the **Clear All Events** button. A confirmation message is displayed. Click the **Yes** button to clear the log.

6. Enter general User Administration parameters using the Policy1 tab.
Settings on this page can be performed only by users with rights assigned by the system administrator.

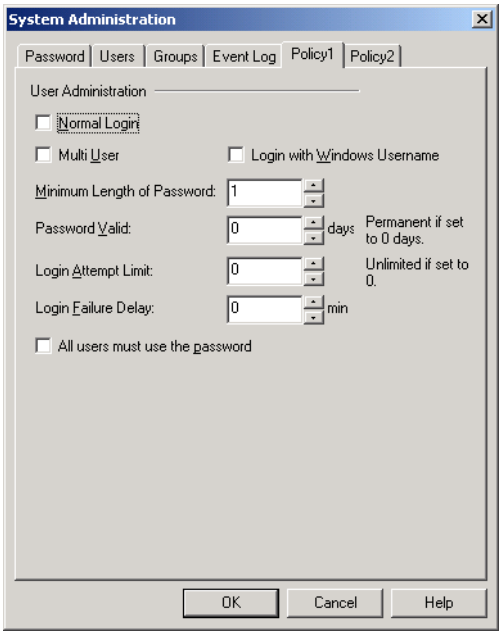


Figure 2.62 System Administration Policy1 Tab

Parameter		Description		Default
Normal Login		Checked	The user name must be entered at login.	
		Unchecked	The "Login" dialog box contains a combo box that lists all of the entered users, and the user name can be selected. By default, the user name that was last logged in is displayed.	Unchecked
Multi User		Unchecked	Only one user may be logged in at a time. After logging in, the GCMSsolution applications are launched without displaying the "Login" dialog box.	Unchecked
		Checked	Multiple users can be logged into the GCMSsolution software simultaneously. Each time a GCMSsolution application is launched, the "Login" dialog box is displayed.	
Login with Windows Username			Available only in the single user mode. The log-in user name for Windows is searched at the start-up of GCMSsolution, and if the same user name is registered for GCMSsolution, the "Login" dialog box is skipped upon users' login.	Unchecked
Password		Minimum Length	Specifies the minimum length of valid passwords from 1 to 8 characters.	1
		Valid	Specifies the validity period of passwords from the time of their creation or alteration. When "0" is set, passwords are permanently effective.	0
Passwords must meet complexity requirements		When selected, passwords consisting of alphabet characters only or numerals only are no longer available. Use a combination of alphabet characters and numerals for passwords.		Unchecked
Lockout Settings (for PC)		If failed login attempts exceed the number specified upon completion of the lockout settings, any user becomes unable to login from that PC for the specified time.		—
	Login Attempt Limit	If login attempts fail more than the set value, the PC to which logins are attempted becomes locked out, and no further login operation will be accepted for the time period set in "Lockout Duration". Specifies the limit of failed login attempts using the [▲] [▼] buttons on the right or entering a value directly. When "0" is set, no limit will be imposed.		0
	Lockout Duration	Enter the time duration to lock out the PC when login attempts fail more than the set value of "Login Attempt Limit". When "0" is set, there will be no PC lockouts.		0
Lockout Settings (for User)		If a user's failed login attempts exceed the number specified upon completion of the lockout settings, that user on the network becomes unable to login for the specified time.		—
	Login Attempt Limit	If login attempts fail more than the set value, the user who attempted logins becomes unable to login for the time period set in "Lockout Duration". Specifies the limit of failed login attempts using the [▲] [▼] buttons on the right or entering a value directly. When "0" is set, no limit will be imposed.		0

Parameter	Description	Default
Lockout Duration	Enter the time duration to lock out the user when login attempts fail more than the set value of "Login Attempt Limit". When "0" is set, the user will not be locked out.	0
# of Levels for Duplicate Password Checking	Prohibits assigning a password which has already been used within the specified times of the recent password change recorded in the history. Setting this parameter to "0" will cancel the duplication check.	0
All users must use password	When selected, any user without a password must set one the next time they open the "User Property" box.	Unchecked

Note

If "SHIMADZU User Authentication Tool" is set in the "User Authentication Mode Select" window at the installation of the GCMSsolution, the policy setting range is determined according to "SHIMADZU User Authentication Tool".

Note

If the length of time for which a password is valid has been exceeded, a message is displayed instructing the user to change the password. Click the **OK** button to display a dialog box for changing the password.

7. The Policy2 tab is used for additional user administration settings.
Settings on this page can be performed only by users with rights assigned by the system administrator.

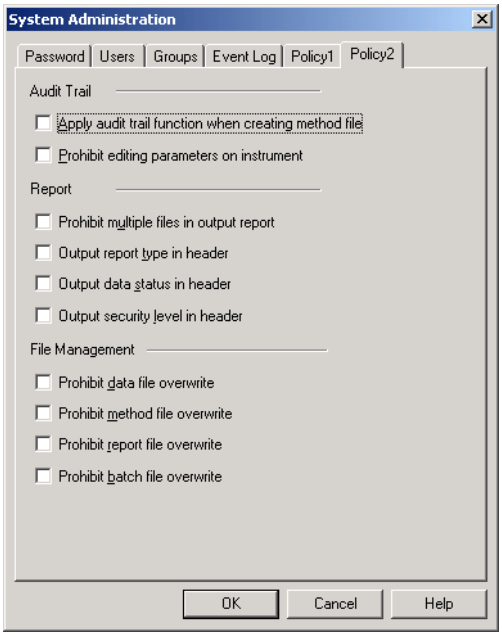


Figure 2.63 System Administration Policy2 Tab

	Setting Item	Description	Default
Audit Trail	Apply audit trail function when creating method file	This forces audit trail settings to be used when a new method is created. It is the same when a new method is created by selecting a template.	Unchecked
	Prohibit editing parameters on instrument	Prevents the user from changing the instrument parameters in the method file. The parameters can be changed in the instrument monitor.	Unchecked
Report	Prohibit multiple files in output report	Prevents loading and printing of files for individual items in the report item Properties File tab.	Unchecked
	Output report type in header	Prints in the header the type of application from which the report is output.	Unchecked
	Output data status in header	Print the data status in the header.	Unchecked
	Output security level in header	Print the security level in the header.	Unchecked
File Management	Prohibit data file overwrite	The file name must be changed when saving to an existing data file during single run and batch run analyses. In the GCMS postrun analysis screen, an existing data file cannot be overwritten.	Unchecked
	Prohibit method file overwrite	In the GCMS analysis and GCMS post analysis data acquisition screen and calibration curve screen, an existing method file cannot be overwritten.	Unchecked
	Prohibit report file overwrite	In the report generation screen of all the applications, an existing report file cannot be overwritten.	Unchecked
	Prohibit batch file overwrite	In the batch table screen of all the applications, an existing batch file cannot be overwritten.	Unchecked

Note

These settings are stored in the "SysAdmin.mdb" database file in the system folder (normally, in C:\GCMSsolution, under a standard installation). In addition, the screen color setting. etc., for individual users are stored in the "SysAdmin.upf" found in the same folder.
As a safety contingency for unexpected accidents or errors, it is recommended that you backup these files.

2.9.6 Templates

If method, batch, or report format files are expected to have similar parameters, templates for each type of file may be created to save time and prevent errors in parameter settings.

1. Creating a Template
Although the procedure below describes how to make a method file template, it applies to batch and report format templates as well.

(1) Enter the method parameters to be included in the template. Select **File > Save Method As Template**. The "Save As Template" dialog box is displayed.

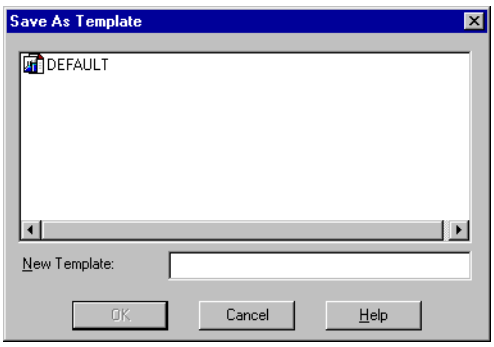


Figure 2.64 "Save As Template" Dialog Box

(2) Enter a name for the template in the New Template text box, and click the **OK** button. If a template with that name does not exist, the template is saved. If a template with the same name does exist, a confirmation message is displayed to verify overwriting the existing file. Click **OK** to overwrite the file and save the template.

2. Generating New Files from Templates

Although the procedure below describes how to generate a new method file from a template, it applies to batch and report format files as well.

(1) Select **File > New Method File** to open the "File New" dialog box.

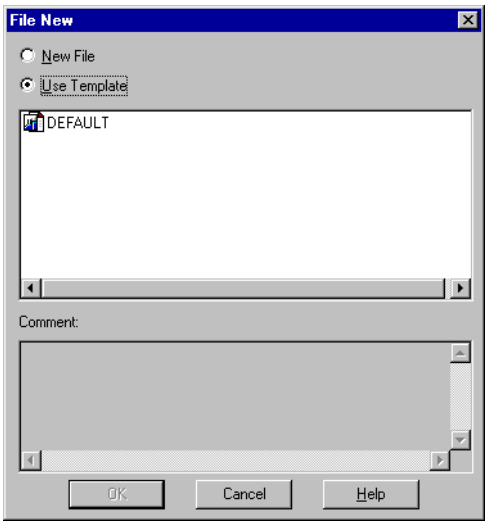


Figure 2.65 "File New" Dialog Box

(2) Select the **Use Template** radio button, and select which template to use from the list box. Click the **OK** button. The new file, created from the content of the template, is displayed.

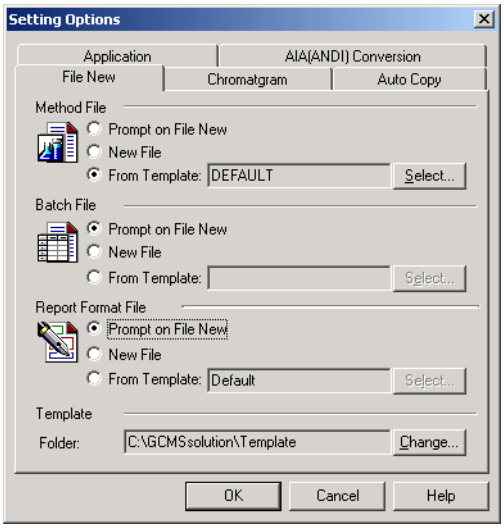


Figure 2.66 "Setting Options" Dialog Box

(3) Click the **Create from Template** option button, select the appropriate template from those listed in the list box, and then click **OK**.
The contents of the template are reflected in the newly created file, and the edit screen is displayed.

Note

Select the New File radio button if the new file should not include content from the template.

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3 Data Acquisition

3.1 Overview

This section describes the windows and functions associated with data acquisition.

3.1.1 "Method Development Acquisition" Window

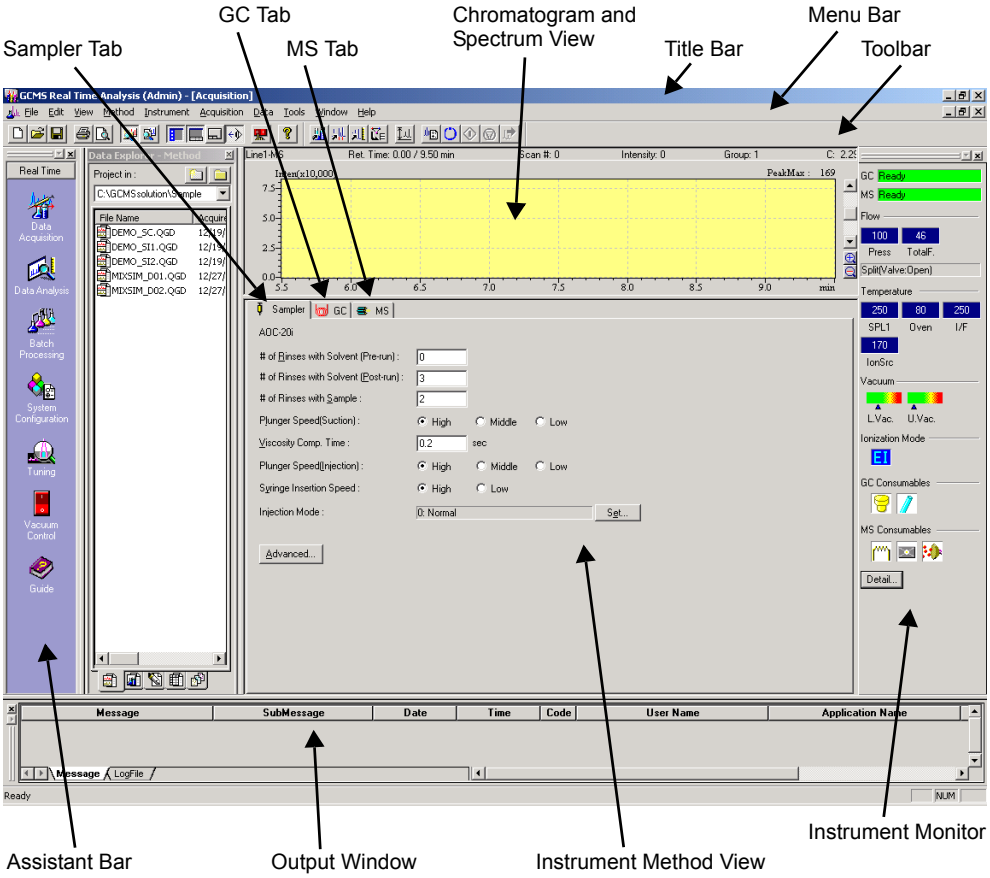

Figure 3.1 "Method Development" Window

This screen layout is displayed when **Method > Instrument Parameters** is selected. It can also be displayed by clicking the **Method Development** icon in the Assistant Bar, or the **Data Acquisition Param** toolbar button. This layout is stored for each user when the "Real Time Analysis" window is closed.
The items in this screen are described in the following table.

"Method Development Acquisition" Window

Title Bar	Displays currently operating application names, process names or method file names.
Menu Bar	Displays various command menus for the displayed window.
Toolbar	Displays buttons for the various command tools for the displayed window.
Assistant Bar	Lists command icons corresponding to the general operation sequence. Ordinarily, the respective operation is selected by clicking these icons.
Chromatogram/Spectrum View	Displays real time data that has been transferred from the detector during data acquisition.
Instrument Method View	Parameters to be used during data collection are set for each unit of the instrument. To modify the parameter settings, click the tab corresponding to the unit.
Instrument Monitor	Displays various instrument parameters in real time. Used to determine whether the instrument is ready to initiate analysis.

3.1.2 "Data Acquisition" Window

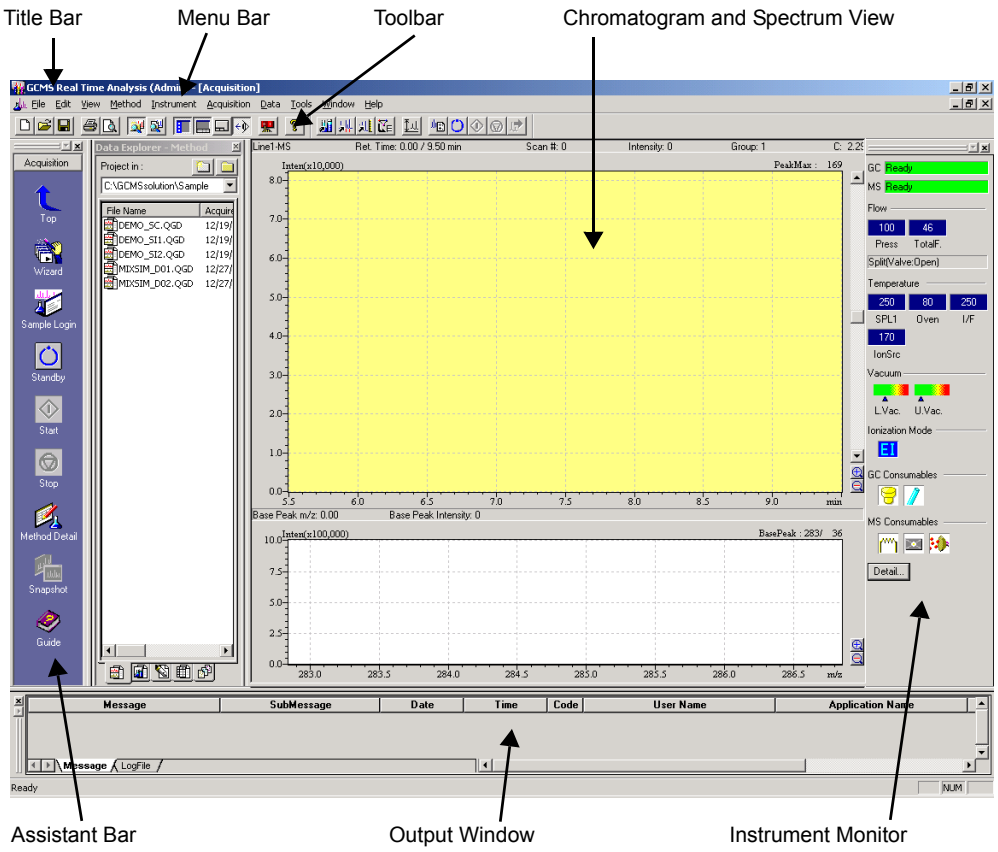

Figure 3.2 "Data Acquisition" Window

This screen layout is displayed when **Data Acquisition** is selected from the Assistant Bar, or when **Instrument Parameters** is deselected from the Method menu or toolbar. The method development mode is exited when the check does not appear next to the Instrument Parameters menu item and the **Data Acquisition Param** toolbar button is not inverted. This layout is stored for each user when the "Real Time Analysis" window is closed.

Title Bar	Displays currently used application names or process names and method file names.
Menu Bar	Displays various command menus of the displayed window.
Toolbar	Displays buttons for the various command tools for the displayed window.
Assistant Bar	Lists command icons corresponding to the general operation sequence. Ordinarily, the respective operation is selected by clicking these icons.
Chromatogram/Spectrum View	Displays real time data that has been transferred from the detector during data acquisition.
Instrument Monitor	Displays various instrument parameters in real time. Used to determine whether the instrument is ready to initiate analysis.

3.1.3 Assistant Bar

This section describes the various icons displayed in the Acquisition Assistant Bar during data acquisition.

 Top	Top	Returns to the Real Time Assistant Bar.
 Wizard	Instrument Method Wizard	Sets instrument parameters for GC, MS, and autosampler used for analysis.
 Sample Login	Sample Login	Used for single runs to assign a file name to the data file obtained by data acquisition, select a vial number and determine whether to print a report.
 Standby	Standby	Downloads the parameters set in the Instrument Parameters view to the instrument, and places the instrument in ready mode, so that data acquisition can be initiated.
 Start	Start	Initiates data acquisition. The retention time is displayed, and the chromatogram or spectrum is displayed in real time.
 Stop	Stop	The MS operation is halted. The GC temperature elevation program and the GC time program continue. The GC operation is stopped if the STOP button on the GC instrument is pressed.
 Instrument Parameters	Instrument Parameters	Display instrument parameters. On/off can be selected.
 Snapshot	Snap Shot	GCMS post run analysis is displayed and MS data from the start of data acquisition to the clicking the **Snap Shot** button can be examined.
 Guide	Guide	Describes the procedure of data acquisition.

3.1.4 Overall Data Acquisition Process

The data acquisition process is performed as described below.

Develop Method	In method development, the instrument parameters of the GC, MS and autosampler are set. When a method has been previously constructed, the content of the method file is checked See Section 3.2 "Method Development Parameters", page 90.
↓	
Login Sample	The sample name, sample ID, the data file name, and other information is entered for a single sample. See Section 3.3 "Single Run Setup", page 104.
↓	
Inject Sample and Acquire Data	The system is prepared for data acquisition, and the sample is introduced manually or automatically. See Section 3.4 "Data Acquisition", page 107. During data acquisition, acquired data can be analyzed. See Section 3.4.3 "Data Analysis", page 108.
↓	
Analysis	Acquired data is saved and analysis is completed. For subsequent data processing, refer to Chapter 4 "Qualitative Analysis" on page 111, Chapter 5 "Quantitative Analysis" on page 137 and Chapter 6 "Generating Custom Reports" on page 165. The tasks of data acquisition through analysis may also be performed automatically. Refer to Chapter 7 "Continuous Analysis" on page 175.

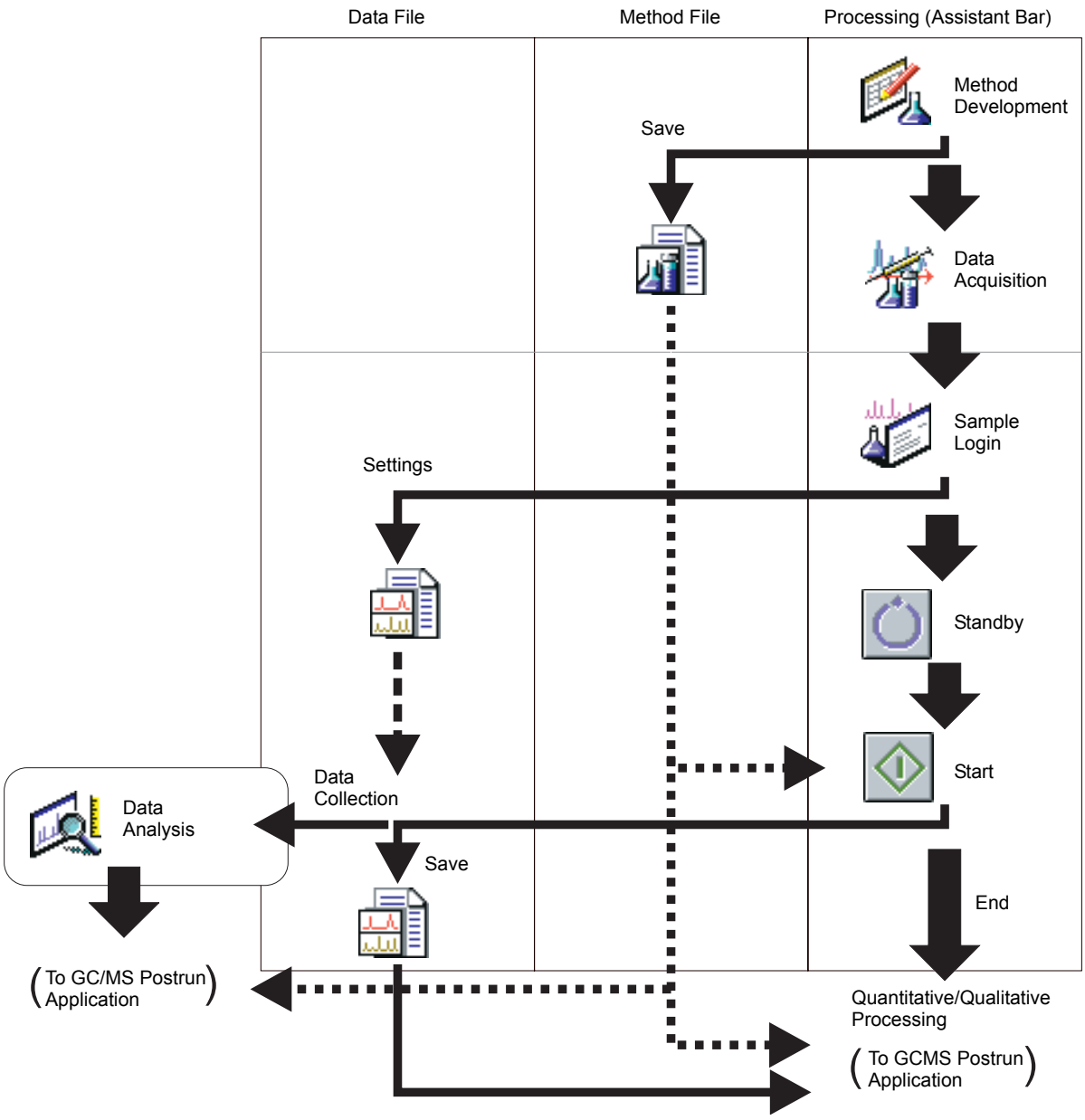

Figure 3.3 The Data Acquisition Process

3.2 Method Development Parameters

This section describes how to develop a Method using the parameters in the "Acquisition" window. When a method is saved as a method file, consistent analysis results can be obtained by using the same method.
Perform system configuration using the GCMS Real Time Analysis application before creating a method. A method can be created in **Instrument Parameters** or by using the **Method** Wizard.

3.2.1 Instrument Parameters

Click the Method wizard icon in the Assistant bar to open the Method wizard window.

(1) Sampler
Displayed when the AOC is selected in system configuration. Refer to Section 3.2.2 "Sampler (Autosampler) Tab", page 91 for parameters and settings. Click the **Next** button to continue.

(2) GC Parameters (1)
Displayed when the GC is selected in system configuration. Refer to Section 3.2.3 "GC Parameters (GC Tab)", page 93 for parameters and settings. Click the **Next** button to continue.

(3) GC Parameters (2)
Displayed when the GC is selected in system configuration. Refer to Section 3.2.3 "GC Parameters (GC Tab)", page 93 for parameters and settings. Click the **Next** button to continue.

(4) MS Parameters (1)
Displayed when the MS is selected in system configuration. Refer to Section 3.2.4 "MS Parameters (MS Tab)", page 97 for parameters and settings. Click the **Next** button to continue.

(5) MS Parameters (2)
Displayed when the MS is selected in system configuration. Refer to Section 3.2.4 "MS Parameters (MS Tab)", page 97 for parameters and settings. When the setting is finished, click the **OK** button. The "Instrument Method Wizard" window closes and a method file is created. If additional settings are required, edit the GC and MS tabs in the "Instrument Parameter" window.

3.2.2 Sampler (Autosampler) Tab

Select **View > Instrument Parameters > Sampler tab**. The Sampler tab is displayed.

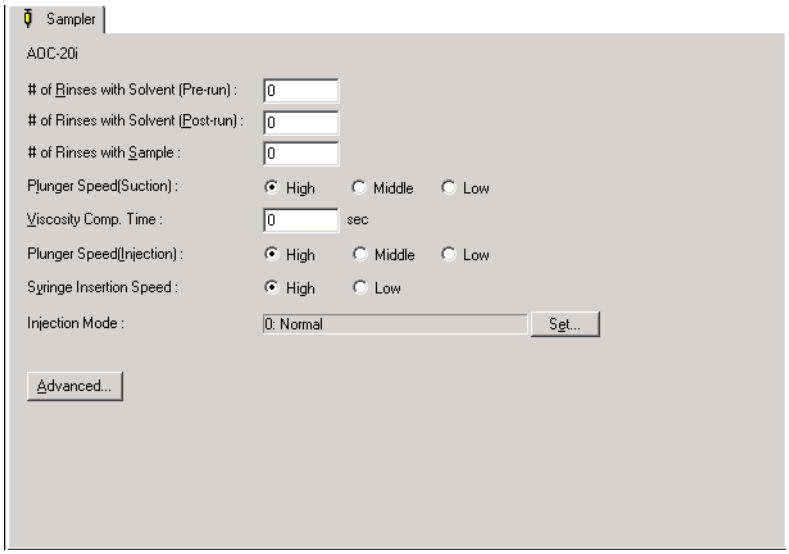

Figure 3.4 Instrument Parameters Sampler Tab

Data acquisition and analysis can be performed with the default values. Refer to the table below when more detailed settings are desired.

Parameter	Description	Unit	Range	Default
# of Rinses with Solvent (Pre-run)	Specifies the number of times the syringe is rinsed with solvent before injection. When Solvent Flush is selected as the injection mode, this setting is not available.		0 - 99	0
# of Rinses with Solvent (Post-run)	Specifies the number of times to rinse the syringe with solvent after injection into the gas chromatograph. Change this setting when a different type of sample is to be injected, or when the same sample is injected multiple times.		0 - 99	1
# of Rinses with Sample	Specifies the number of times the syringe is rinsed with sample before injection. Change this setting when a different type of sample is to be injected, or when the same sample is injected multiple times.		0 - 99	2
Plunger Speed (Suction)	Specifies the plunger speed when aspirating a sample. This speed applies to sample aspiration for sample rinsing and for sample injection. For pumping and solvent rinses, the aspiration speed is always High speed.		High, Middle, Low	High
Viscosity Comp. Time	Specifies the wait time between aspiration and injection. Low viscosity liquids are drawn up into the syringe simultaneously with the plunger. A longer wait time should be specified if high viscosity liquids are used.	sec.	0 - 99.9	0.2

Parameter	Description	Unit	Range	Default
Plunger Speed (Injection)	Selects the speed of the plunger during sample injection. This is not the speed at which the syringe descends into the injection port.		High, Middle, Low	High
Syringe Injection Speed	Selects the speed at which the syringe descends into the injection port.		High, Low	High
Injection Mode	Selects Normal or one of the Solvent Flush modes for the injection. Press the **Set** button to change the injection mode.			0
Advanced	Allows making more detailed settings.			
Injection mode	Sets the injection mode. Setting is performed by clicking on the **Setting...** button to open the "Injection Mode" window. Select from among the following 5 options.		0 - 4 3: Can be selected when equipped with AOC-20s. 4: Can only be selected when equipped with AOC-20s.	0
	0: Normal injection (Sample only)			
	1: Sample + air + solvent			
	2: Sample + solvent			
	3: Sample + air + Standard + air + solvent			
	4: Sample + Standard + solvent			
	When an injection mode other than normal injection is selected (i.e. solvent flush mode), select one of the following two options to set number of number of solvent rinses prior to sample injection. Same as number of sample rinses prior to injection Same as number of solvent rinses after injection After making the setting, click the **OK** button to complete the settings.			

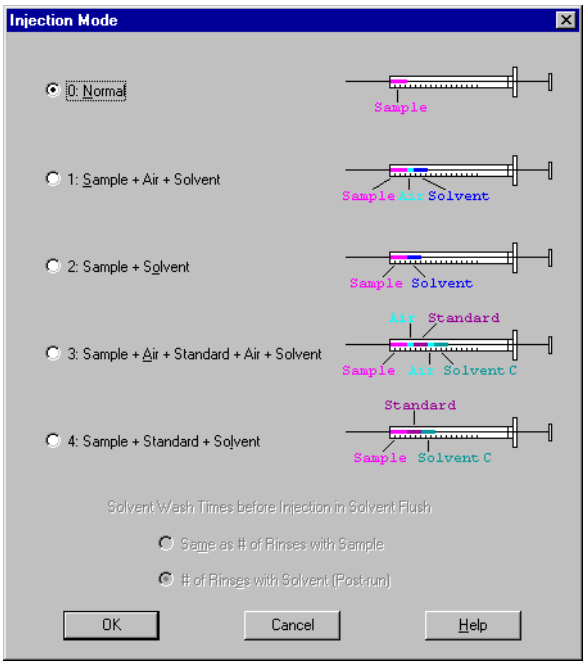

Figure 3.5 "Injection Mode" Dialog Box

Note

When the **Advanced** button is selected, more detailed autosampler parameters are provided. Refer to GCMS Help for further information on specific parameters.

3.2.3 GC Parameters (GC Tab)

This section describes how to set the parameters for the gas chromatograph.
Select **View > Instrument Parameters > GC tab**. The GC tab is displayed.

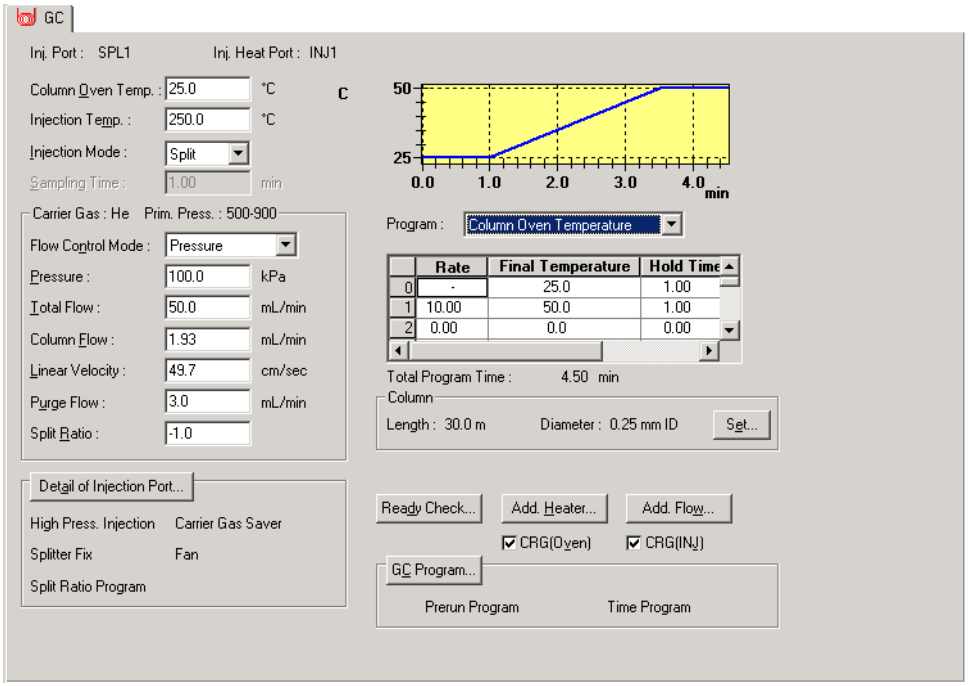

Figure 3.6 Instrument Parameters GC Tab

Note

The Control Mode parameter determines which Carrier Gas parameters will be displayed. The Carrier Gas parameters include Column Inlet Pressure, Column Flow, Linear Velocity, Split Ratio, Total Flow, and Carrier Gas Flow. Pressure and flow control parameters cannot be set for an instrument that does not have an electronic flow rate controller.

Note

If the wide bore column (internal diameter 0.53 mm) is used, the flow rate control range at low oven temperature is narrowed, and may cause a flow rate control error at the GC. Care must be taken when setting the flow, using the following guidelines.

Control Ranges (using full volume injection with column I.D. 0.53 mm, length 30 m)

Oven Temperature (ºC)	Flow Rate Setting Range (mL / min)
50	10 - 15
100	7 - 15
150	6 - 15
200	4 - 15

Depending on the flow rate, an error may be generated when the oven temperature is lowered even if it is running normally at a higher temperature.

Parameter	Description	Unit	Range	Default
Column Oven Temp.	Specifies the temperature of the column oven.	°C	–99 - *	50
Injection Temp.	Specifies the injection port temperature.	°C	–99 - *	25
Injection Mode	Selects the injection mode. This parameter is not displayed for OCI/WBI.		Split/ Splitless/ Direct	Split
Sampling Time	If Splitless is selected as the Control Mode, the Sampling Time parameter specifies the interval between the time of sample injection and the time at which the split flow path is opened.	min.	0 - 9999.99	1
Flow Control Mode	Selects the control mode of carrier gas flow. Flow can not be selected when the injection mode is Split or Splitless.		Flow/Linear Velocity/ Pressure	N/A in Split mode
Pressure	This parameter is disabled when Direct is selected in the Injection Mode and Flow is selected in the Flow Control Mode.	kPa	0 - 970	100 kPa
		kgf/cm^2	0 - 9.89	
		psi	0 - 140.7	
Total Flow	Total Flow = Column Flow + Split Flow + Purge Flow. The value is held constant during the programmed temperature gas chromatography. This parameter is disabled when Direct is selected in the Injection Mode and Linear Velocity or Pressure is selected in the Flow Control Mode.	mL/min	0 - 1200	50
Column Flow	Displays the rate of volumetric flow inside column. The Column Flow is automatically calculated from the Column Inlet Pressure. When the Model is set to "Dual TMP" in the System Configuration window, it is recommended that the Column Flow parameter be set to 15 mL/ min or below. It is also recommended that this parameter be set to 2 mL/min or below for Single TMP models. This is because an insufficient degree of vacuum is obtained when exceeding these values, and may have a bad influence on ion generation.	mL/min		

Parameter	Description	Unit	Range	Default
Linear Velocity	Displays the linear velocity. Linear velocity is automatically calculated from the Column Inlet Pressure.	cm/sec		
Purge Flow	Specifies the purge flow. Disabled in the case of No Purge APC.	mL/min	0.0 - 1200	
Split Ratio	Specifies the split ratio or the ratio of the column flow to the split flow. When a split ratio is set, the system sets the total flow rate based on the calculated carriergas flow rate, so that the desired split ratio occurs at the oven temperature. Set the split ratio to –1.0 to fix the total flow rate regardless of the oven temperature. This parameter is disabled when Direct is selected in the Injection Mode parameter.		–1, 0 - 120000	–1
Detail of Injection Port	Opens the "Detail Setting of Injection Port" window. Characters appear in black when details are set. This parameter is disabled for WBI.			
Program	Allows the entry of time program steps. Item to be programmed (oven temperature, pressure, and/or flow rate) can be selected from the drop-down list.		Oven Temperature, Flow, Pressure, Purge Flow	
Rate	The ratio of increase or decrease for a temperature, pressure and flow rate program, expressed in incremental units.			
Temp./Pressure/ Flow	The temperature, pressure, or flow value in a program.			
Hold Time	The amounts of time to maintain a temperature, pressure, or flow rate in a program.	min	0 - 9999.99	
Column	Displays the column related parameters set in System Configuration. To change these parameters, click the Set button in the column section.			
Automatic Zero Correction after Ready	Performs the zero correction automatically when the instrument is ready. For detectors other than MS.			
CRG (Oven)	Uses CRG for the column oven. This parameter is displayed when CRG (Oven) is checked in the Option tab in GC2010 / System Configuration window.			
CRG (INJ)	Uses CRG for cooling of the injection port.This parameter is displayed when CRG (INJ#2) is checked in the Option tab in GC2010 / System Configuration window.			

* Refer to GCMS Help for upper limits.

To change the parameters for a time-controlled GC during data acquisition, create a time program. Click the **GC Program** button to open the "GC Program" dialog box.

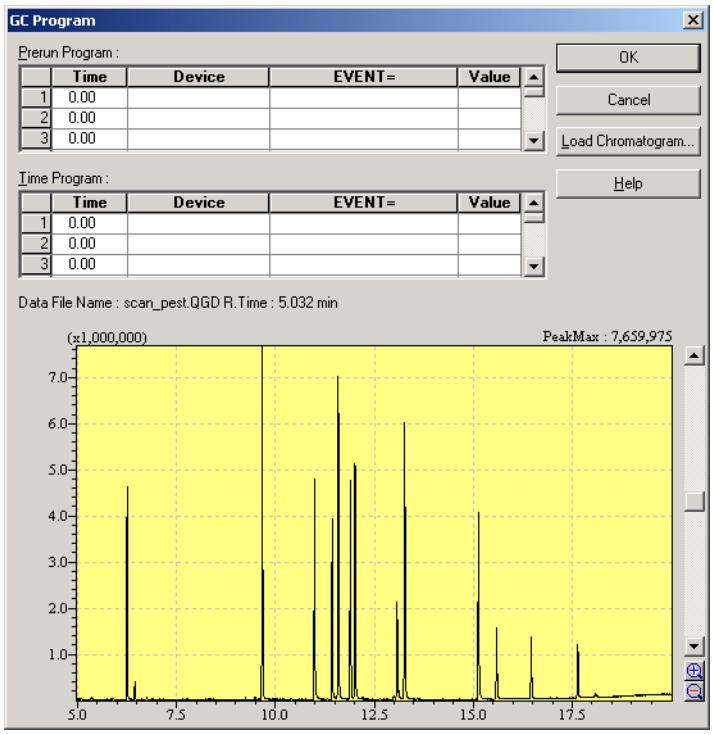

Figure 3.7 "GC Program" Dialog Box

A pre-run program is a time program that controls the operation of various units prior to analysis.

A run-time program is a time program that controls the operation of various units during analysis.

Parameters	Description	Units	Range
Time (min)	Enters the retention time at which to perform the command.	min.	0 - 655
Device	Selects the unit to be used from the list in the combo box. Devices are defined in the Instrument Configuration menu.		
Event	Select the appropriate command. Refer to GCMS Help for more information.		
Setting	Enters a value that corresponds with the selected device and event. Refer to GCMS Help for more information.		

Note

When the **Load Chromatogram** button of the "GC Program" dialog box is selected, the "GCMS Data File Open" dialog box opens, and the TIC can be read from an existing data file. Click the chromatogram while one of the Time (min) fields is selected to enter that retention time into the selected Time (min) field.

3.2.4 MS Parameters (MS Tab)

This section describes the parameters for the MS. Select the **Method > Instrument Parameters > MS tab** to view the MS parameters.

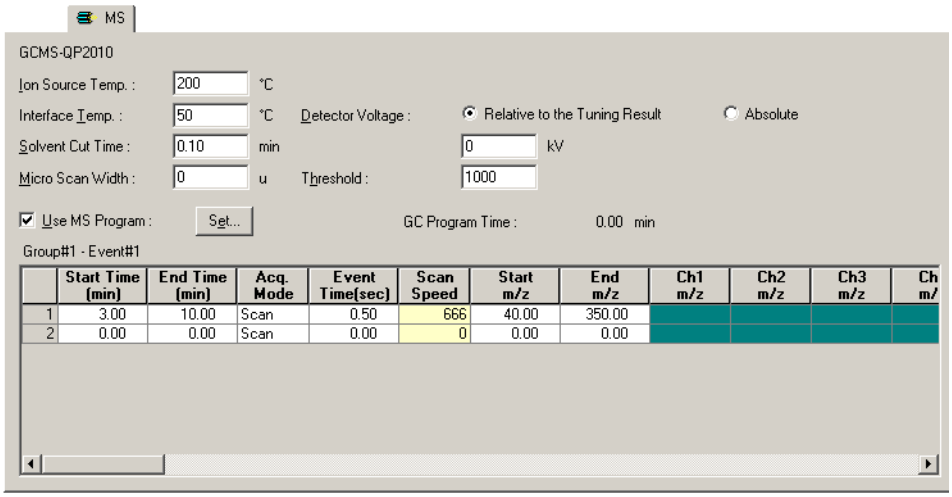

Figure 3.8 Instrument Parameters MS Tab

Data acquisition and analysis can be performed with the default values. Refer to the table below when more detailed settings are desired.

Parameter	Description	Unit	Range	Default
Temperature	Enters the temperature of the ion source.	ºC	100 - 260	Values from System Configuration
Interface Temperature	When GC is connected, temperature of the GC heat port connected to MS is definable. When GC is not connected, interface temperature appears in gray.	ºC	0 - *	25
Solvent Cut Time	Sets the time to turn on the filament once the solvent elutes after sample injection. Normally, when the sample is injected, a large quantity of solvent is introduced into the analysis system. This results in a sharp decrease in the vacuum inside the ion source, which has a detrimental effect on the filament and other components. To prevent this, the filament is turned OFF while the solvent is passing through the analysis system. ***Note:*** When analyzing a sample with peaks that elute before the solvent, use an MS program to turn the filament ON and OFF.	min.	0 - 9999.9	2.00
Detector Voltage	Sets the detector voltage. Selecting the radio button determines whether to handle the setting in the Edit box as an absolute value or as a relative value. When "Absolute" is changed to "Relative," the gain is set to 0. Absolute: Used when directly setting the voltage. Relative to the Tuning Result: Used when setting an absolute value from the voltage in the tuning file to be used. Positive (+) and negative (-) symbols may be used. ***Caution:*** Note that when the voltage is set at a high value, a larger number of ions will be detected and the detector could be damaged. However, as the instrument ages and sensitivity drops, the detector voltage can be gradually increased.	kV	Abs: 0.50 - 3.00 Rel.: -2.50 - 2.50	Rel. 0
Threshold	Sets the noise threshold level. Ion signals below this value will be treated as noise and deleted from the data. This parameter is setable when Scan is selected in Acquisition Mode.	count	0 - 9999	1000

Parameter	Description	Unit	Range	Default
Use MS Program	Specifies whether to use an MS program. When checked, the **Set** button is enabled.			
Micro Scan Width	Sets the scan width when performing micro scan measurement. The Setting range is (0.00 to 1.00). When set to "0," micro scanning is not performed. About Micro Scan Measurement: In SIM analysis, data is collected with the MS control fixed at a set m/z value. Micro scanning is a measurement method that collects data with the MS scanning over a minute range. Improved reproducibility can be expected in the analysis data when this method is used.	amu	0.00 - 1.00	0

* Refer to GCMS Help for upper limit.

The parameters below differ, depending on whether the Acquisition Mode is Scan or SIM.

Scan Parameters

	Start Time (min)	End Time (min)	Acq. Mode	Event Time(sec)	Scan Speed	Start m/z	End m/z
1	3.00	10.00	Scan	0.50	666	40.00	350.00
2	0.00	0.00	Scan	0.00	0	0.00	0.00

Figure 3.9 Scan Parameters

Parameter	Description	Unit
Start Time	Sets the data acquisition start time.	min.
End Time	Sets the data acquisition end time.	min.
Acquisition mode	Selects scan.	
Event Time	Sets the interval for a single scan.	Sec.
Start m/z	Sets the starting mass range m/z.	u
End m/z	Sets the ending mass range m/z.	u
Scan Speed	Sets the speed at which scanning is performed in the specified m/z range. This will be automatically selected and displayed from the possible settings according to the measurement m/z range and the Event Time. A smaller value (slower scan) reduces the noise in the data.	

SIM Parameters

	Start Time (min)	End Time (min)	Acq. Mode	Event Time(sec)	Scan Speed	Start m/z	End m/z	Ch1 m/z	Ch2 m/z	Ch3 m/z	Ch4 m/z
1	3.00	10.00	SIM	0.20				298.30	191.00	141.00	156.50
2	0.00	0.00	SIM	0.20				0.00	0.00	0.00	0.00

Figure 3.10 SIM Parameters

Parameter	Description	Unit
Start Time	Sets the data acquisition start time.	min.
End Time	Sets the data acquisition end time.	min.
Acquisition mode	Selects SIM.	
Event Time	Sets the interval for a single scan.	Sec.
Ch1-m/z - Ch64-m/z	Sets the m/z for each channel.	u

FASST (Fast Automated Scan/SIM Type) Paramenters

	Start Time (min)	End Time (min)	Acq. Mode	Event Time(sec)	Scan Speed	Start m/z	End m/z	Ch1 m/z	Ch2 m/z	Ch3 m/z	Ch4 m/z
1	3.00	10.00	Scan	0.50	666	40.00	350.00				
	3.00	10.00	SIM	0.20				298.30	191.00	141.00	156.50
2	0.00	0.00	Scan	0.00	0	0.00	0.00				

Figure 3.11 If both Scan and SIM are selected.

If the Start Time and End Time are set to the same time respectively in successive two rows, the data of the two rows are acquired every Event Time one after the other. In this case, the first row is called Event 1, and the second row Event 2.
The Group number is displayed in the left column of the table. If two events are set for a group, the Group number is displayed in the row of Event 1 only. The event number and group number for each row is displayed in the top left of the table as "Group Number-Event Number".
The maximum number of events for a group is 2. Set the Acquisition mode for Event 1 to Scan, and SIM for Event 2.

For details, see FASST Measurement Operation Manual.

Note

Up to 128 groups can be set.
However, with a version of GCMSsolution earlier than version 2.40, if a method file containing more than 65 groups is opened, it may not run normally. If the instrument parameters of a data file with more than 65 groups are displayed, it will not run normally. It will not be able to display chromatograms for groups after the 65th group.

To change the MS parameters during data acquisition, use a time program. Select the Use MS Program check box, and then click the **Set** button to open the "MS Program" dialog box.

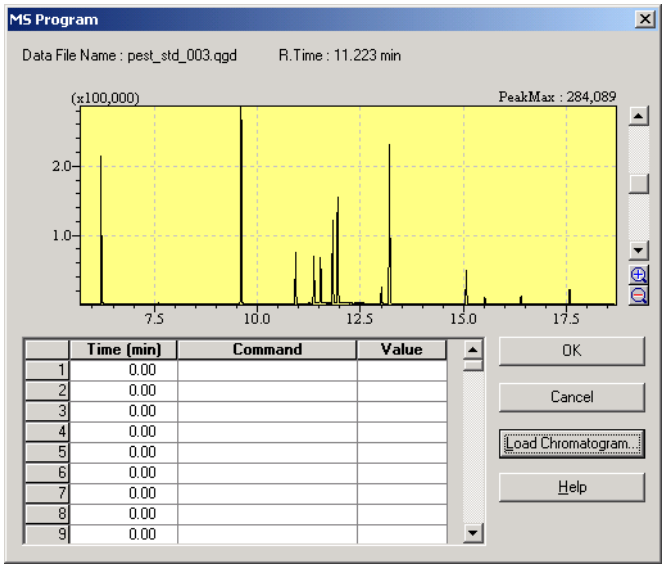

Figure 3.12 MS Program "Dialog Box"

Enter the parameters into the MS Program table as described below.

Parameter	Description		Unit	Range
Time (min)	Specifies the retention time at which to perform the command.		min.	
Command	Selects a command to execute during data acquisition. Click the desired cell, and the cell becomes a combo box. Select a command from the combo box.			
	DetectorVolts=	Changes the detector voltage at the specified time.	kV	0.50 - 3.00
	FilamentON	Turns on the filament at the specified time.		No value.
	FilamentOFF	Turns off the filament at the specified time.		No value.
	PFTBAOpen	Opens the standard (PFTBA) solenoid valve at the specified time.		No value.
	PFTBAClose	Closes the standard (PFTBA) solenoid valve at the specified time.		No value.
	Reagentgas1Open	Opens the reagent gas1 solenoid valve.		No value.
	Reagentgas1Close	Closes the reagent gas1 solenoid valve.		No value.
	Reagentgas2Open	Opens the reagent gas2 solenoid valve.		No value.
	Reagentgas2Close	Closes the reagent gas2 solenoid valve.		No value.
Value	Specifies a value for Command parameters that require one.			

Note

When the **Load Chromatogram** button of the "MS Program" dialog box is selected, the "GCMS Data File Open" dialog box opens, and the TIC can be read from an existing data file. Click the chromatogram while one of the Time (min) fields is selected to enter that retention time into the selected Time (min) field.

3.3 Single Run Setup

This section explains how to set the parameters for a single run, including sample name, sample ID, data file name, and sampler parameters, if used.

3.3.1 "Sample Login" Dialog Box

Select the **Sample Login** icon of the Data Acquisition Assistant Bar. The "Sample Login" dialog box opens.

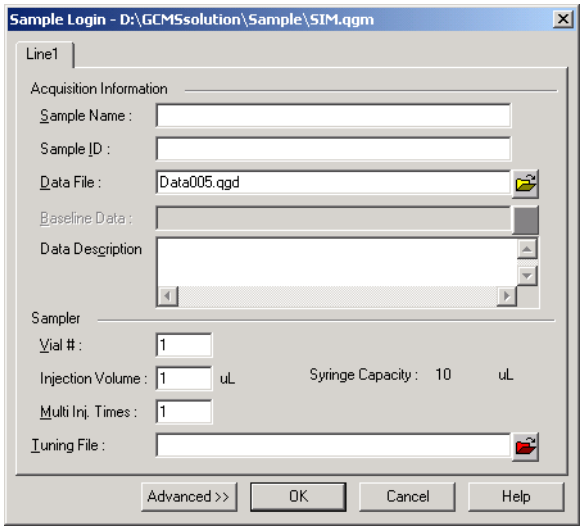

Figure 3.13 "Sample Login" Dialog Box

Sample Name	Specifies the name of the sample. The name is used for identifying data files and is output on reports.
Sample ID	Specifies the ID number or lot number of the sample. It is useful to set this item since it can be used as a search parameter for the data file. It can be changed during data acquisition. Up to 31 characters may be entered.
Data File	Specifies the filename for saving the acquired data. The file extension is automatically added. The path of the file when only the name was entered results in the same path as the method file opened upon Data Acquisition.
Base Line Data	Sets a data file name for differential chromatogram (base line chromatogram). This field is only used when GC detector is used.
Data Description	Specifies comments to be registered in the datafile.

The "Select Data File" dialog box opens when the **Folder** button to the right of the text field is clicked. Select a directory in which to save the data and enter the file name.

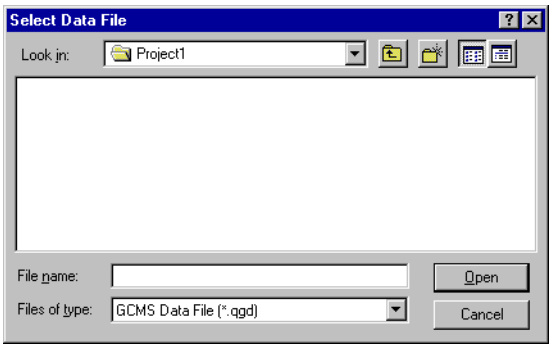

Figure 3.14 "Select Data File" Dialog Box

Note

If the "Sample Login" dialog box is used to analyze a sample multiple times, or if the file name that has been entered is the same as an existing file, confirm whether to overwrite the file when the analysis begins. Add a number to the file name and use the automatic increment function in a batch table to avoid overwriting data. Check for duplicate file names prior to initiating data acquisition.

Sampler Parameters (See Figure 3.13 "Sample Login" Dialog Box)

Vial #	Specifies the position occupied by the vial in the autosampler.
Injection Volume	Specifies the amount of sample to inject. This volume is injected the number of times set in Multi Inj. Times.
Multi Inj. Times	Specifies how many times to inject the sample.
Tuning File	If the Tuning File field is left blank, the tuning file either used during the last data acquisition or saved during the last tuning is used. The path of the file when only the file name is entered is GCMSsolution\System\Tune#. (# is the system number; GCMSsolution is the folder specified during the installation.)
Syringe Capacity	Verifies the syringe capacity specified in System Configuration. Note that this parameter cannot be edited from the "Sample Login" dialog box.
Report	Selects the printing of a report after data acquisition is completed. Enter the full file name of the report format file to be used to print the report, or click the **File Folder** button to browse for the file. The "Select Report Format File" dialog box is displayed. Select the file, and click the **Open** button. The full path is now displayed in the Report text box. The path of the file when only the name was entered results in the same path as the method file opened upon Data Acquisition.

The "Select Report Format File" dialog box opens when the **Folder** button to the right of the Report text field is clicked. Open the directory in which file is saved, and select the file name.

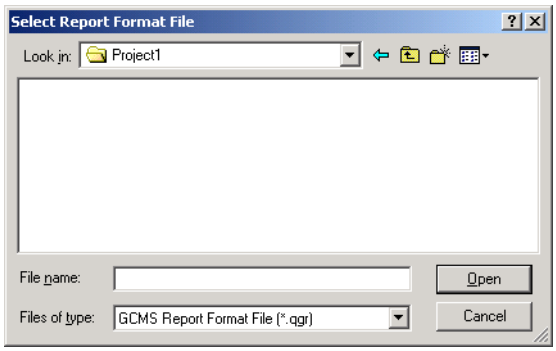

Figure 3.15 "Select Report Format File" Dialog Box

For a description of the Sample Login Advanced parameters, refer to GCMS Help.

3.4 Data Acquisition

This section describes instrument preparation and data acquisition. During acquisition, real time data is displayed, and upon completion, it is saved as a data file.

3.4.1 Standby

During Standby, the method is downloaded to the instrument and the instrument is prepared for analysis.

Select the **Standby** icon on the Data Acquisition Assistant Bar.

Note

When the **Standby** icon is selected, and the method has not been saved, a dialog box is displayed to prompt the user to save the method.
To save the method, select the **Yes** button. If the method has been saved previously, the old method file is overwritten with the new information. If the method has not been saved, the "Save Method File As" dialog box is displayed. Select a directory, enter a filename, and click the **Save** button. The method is then downloaded to the instrument.
To download the method to the instrument without saving it, select the **No** button. The method is downloaded to the instrument.
To abort the method download, select the **Cancel** button.

3.4.2 Start

Select the **Start** icon in the Data Acquisition Assistant Bar. Acquisition and analysis are performed as specified in the selected method.

The real time spectrum and chromatogram are displayed.

Note

Selecting the **Start** button on the GC unit can also start data acquisition, whether an autosampler is used or not.

3.4.3 Data Analysis

During data acquisition, the acquired data is written to the data file. If the GCMS Postrun Analysis application is launched by selecting the **Snap Shot** icon in the Data Acquisition Assistant Bar, the acquired data is displayed.

Note

For more information about the GCMS Postrun Analysis application, refer to Chapter 4 "Qualitative Analysis" on page 111 and Chapter 5 "Quantitative Analysis" on page 137.

3.4.4 Stop

Data acquisition and analysis may be stopped during a run. Select the **Stop** icon in the Data Acquisition Assistant Bar.

Caution

If data acquisition and analysis are stopped with the Stop command, the MS program stops immediately, but the GC temperature program does not stop until the sample is processed. This prevents leaving sample residue in the column. To stop the GC immediately, press the Stop button on the GC unit.

3.5 Instrument Monitor

In this section, procedures for monitoring the current status of the instrument are described. Click the Detail button in the Instrument Monitor section to set values and initialize use time for consumable parts.

3.5.1 Instrument Monitor

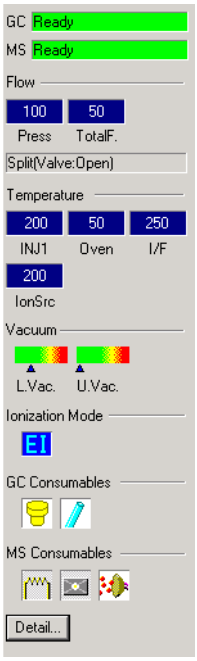

Figure 3.16 Instrument Monitor

Items	Description
GC, MS, HS	Appears in different colors depending on the current status of GC, MS, HS. Refer to GCMS Help for more information.
Units	Select the radio button for Line 1 or Line 2 to display the information corresponding to that unit. These buttons are only displayed if 2 lines are connected.
Vacuum	Indicates vacuum with a graph. Triangle indicators move according to actual values. When the Ion Gauge is set to "None" in the System Configuration window, high vacuums are not displayed.
GC Consumables, MS Consumables	The background color of the icons changes to indicate maintenance or replacement is required.
Other Parameters	To set other parameters, click the **Detail...** button and open the "Instrument Monitoring Details" window. For each parameter, refer to GCMS Help.

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4 Qualitative Analysis

4.1 Overview

This section explains how to use the GCMS Postrun Analysis application for performing qualitative analysis on previously acquired data and printing reports.

4.1.1 "Qualitative Data Analysis" Window

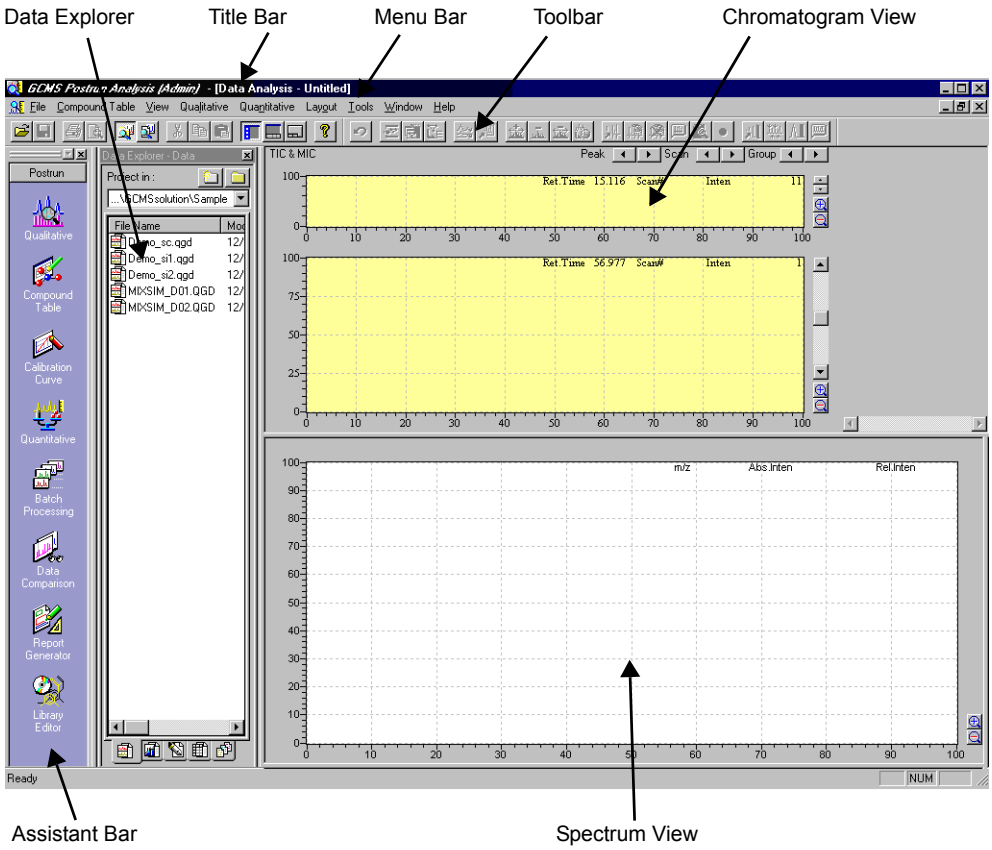


Figure 4.1 "Qualitative Data Analysis" Window

Title Bar	Displays the name of the application, user, active window, and file.
Menu Bar	Displays the menus that are available in the active window.
Toolbar	Displays the command buttons that are available in the active window.
Assistant Bar	Displays the command icons that apply to a specific function or window. To open the window or activate the command, click the appropriate icon.
Data Explorer	Displays all files by type, including Data, Method, Report Format, Batch, and All Files. Data can easily be opened for analysis by double clicking the file icon or dragging it to the appropriate location.
Chromatogram View	Displays the chromatogram from the current data file. All of the acquired data is displayed in the upper TIC window. The TIC, MIC and MC are displayed in the lower MC window.
Spectrum View	Displays the mass spectrum for the current retention time.

4.1.2 Qualitative Analysis Procedures

Procedures for qualitative analysis are described below.

Method Development	The instrument parameters for the GC and MS, and the qualitative, quantitative, data view, and QA/QC parameters, are set in the method file. Create a new method file using GCMS Real Time Analysis, or use an existing method file after verifying its contents. For more information about method development, see Section 3.2 "Method Development Parameters", page 90.

↓

Sample Injection and Data Acquisition	Data is acquired in GCMS Real Time Analysis. Enter information about single samples in the "Data Acquisition" window, or enter information about multiple samples in the "Batch Processing" window. Initiate single or batch processing. For more information about data acquisition, refer to Section 3.3 "Single Run Setup", page 104 and Section 3.4 "Data Acquisition", page 107.

↓

Data Analysis	Open GCMS Postrun Analysis, and click the Assistant Bar **Qualitative Analysis** icon. This chapter describes performing qualitative analysis and reporting.

1. Viewing Acquired Data

Open GCMS Postrun Analysis, and click the **Qualitative Analysis** icon in the Assistant Bar.

The "Data Analysis" window is displayed in qualitative analysis mode.

Click the **Data** tab of the Data Explorer, and double-click the appropriate data file.

The acquired data is displayed in the Chromatogram View.

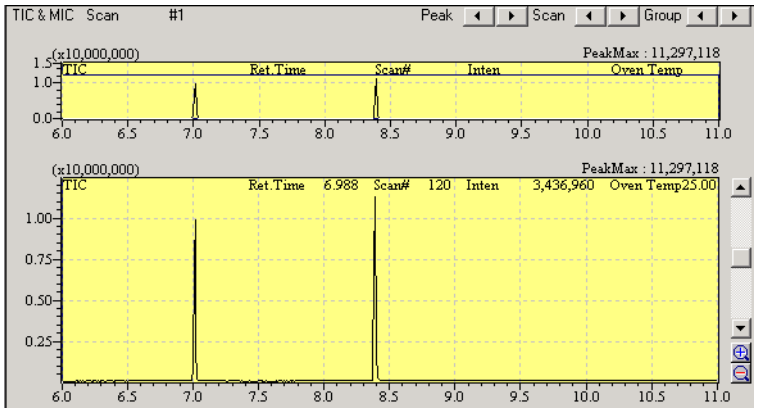


Figure 4.2 Chromatogram View

2. Peak Integration
   Peaks must be integrated before spectra can be chosen for similarity searches. Peaks can be integrated manually or automatically. Refer to Section 4.3 "Peak Integration", page 118.
3. Similarity Search
   The Similarity Search tries to match spectra from the integrated peaks to spectra belonging to known compounds in the library file. Candidates with varying degrees of similarity are displayed for each spectrum in a list. Select a candidate to identify a component of the sample by comparison between the known compound and sample spectra. Refer to Section 4.4 "Similarity Search", page 130.
4. Printing a Qualitative Report
   Using a similarity search, the target compound is compared with candidates from the library; three types of reports about the candidates found by the search can be printed. Refer to Section 4.5 "Printing Results", page 134.

4.2 Spectrum Display

This section explains how to display the mass spectrum for any retention time on a chromatogram.

4.2.1 Displaying the Spectrum

1. To display a spectrum that corresponds to a specific retention time, double-click at the appropriate position on the chromatogram. The spectrum is displayed in the Spectrum View.

2. To display a spectrum that corresponds to a compound that is registered in the Compound Table, select the appropriate row in the table. If quantitation has been performed and the compound was identified, the spectrum is created from the peak retention time set in the Spectrum Process tab of the "Qualitative Parameters" dialog box. The spectrum is then displayed in the Spectrum View.

3. To display a spectrum that is registered in the Spectrum Process Table, select the appropriate row in the table. The spectrum is displayed in the Spectrum View.

4. To display a spectrum from qualitative peak integration data, select the appropriate row in the Qualitative Peak Table. The spectrum is created from the peak retention time set in the Spectrum Process tab of the "Qualitative Parameters" dialog box. The spectrum is then displayed in the Spectrum View.

Note

If the retention time is in FASST measurement interval, the Scan spectrum is displayed.

4.2.2 Averaging the Spectrum and Background Processing

1. To obtain a clean spectrum, average a range of mass spectra. First, turn on the Average function in the Chromatogram View by selecting **View > Spectrum Calculation > Average,** or by right-clicking the chromatogram and selecting **Spectrum Calculation > Average**. With the Chromatogram View in the averaging mode, drag the mouse from the starting to ending retention times to set the range you wish to average. The averaged spectrum is displayed in the Spectrum View. Averaging around the peak apex is usually the most effective.

2. To subtract the background spectrum, first display an original or averaged spectrum. Then turn on the Subtract function in the Chromatogram View by selecting **View > Spectrum Calculation > Subtract**, or by right-clicking the chromatogram and selecting **Spectrum Calculation > Subtract**. With the Chromatogram View in the subtraction mode, determine a retention time representative of the background and double-click it. The spectrum displayed in the Spectrum View is the difference between the original or averaged spectrum and the background spectrum.

An averaged background spectrum can also be subtracted. First display an original or averaged spectrum. Then turn on the Subtract Averaged Spectrum function in the Chromatogram View by selecting **View > Spectrum Calculation > Subtract Averaged Spectrum**, or by right-clicking the chromatogram and selecting **Spectrum Calculation > Subtract Averaged Spectrum**. With the Chromatogram View in the average subtraction mode, determine a range of retention times representative of the background and drag the mouse from the starting to ending retention times to set the range. The spectra for the time range are averaged, then subtracted as the background from the spectrum currently displayed in the Spectrum View. A range around the start or end of the peak is typically used as the background.

3. To subtract background spectrum repeatedly, select **View > Spectrum Calculation > Subtract** command. With the Chromatogram View in the subtraction mode, determine a retention time representative of the background and double-click it while holding down the [Shift] key. Then the subtracted result is displayed without clearing the previous result. Or select the **Spectrum Calculation > Subtract Averaged Spectrum** command from the menu, determine a range of retention times representative of the background and drag the mouse from the starting to ending retention times to set the range. The averaged spectrum is subtracted in the range defined by dragging without clearing the previous result.

Note

If the spectrum averaging is performed in FASST measurement interval, the Scan spectrum is used.
If the spectra to be subtracted is acquired using FASST, the Scan spectrum is used for the subtraction.

Note

The toolbar includes **Average Spectrum**, **Subtract Spectrum**, and **Average & Subtract spectrum** buttons for easier access to the spectrum calculation functions. Use the three spectrum calculation buttons on the toolbar as follows.

 Average Spectrum Button	Click the button, and drag the mouse from the starting to ending retention times to set the range you wish to average. The averaged spectrum is displayed in the Spectrum View.
 Subtract Spectrum Button	Click the button, determine a retention time representative of the background, and double-click it. The spectrum displayed in the Spectrum View is the difference between the original or averaged spectrum and the background spectrum.
 Average & Subtract Spectrum Button	Click the button, determine a range of retention times representative of the background, and drag the mouse from the starting to ending retention times to set the range. The spectrums for the time range are averaged, then subtracted as the background from the spectrum currently displayed in the Spectrum View.

4.2.3 Displaying Results of Spectrum Calculations

The retention time range used to calculate the resultant spectrum is displayed in the top of the Spectrum View. See the table below for simple examples of how the time ranges are displayed.

[10.000]	Spectrum at 10.000 min retention time.
[10.000 -> 10.100]	Resultant spectrum averaged from retention time range of 10.000 min to 10.100 min.
[10.000] - [10.500]	Resultant spectrum when spectrum at 10.500 min retention time is subtracted from spectrum at 10.000 min.
[10.000 -> 10.100] - [10.500]	Resultant spectrum where spectrum at 10.500 min retention time is subtracted from spectrum averaged from retention time range of 10.000 min to 10.100 min.
[10.000 -> 10.100] - [10.500 -> 11.000]	Resultant spectrum where spectrum averaged from retention time range of 10.500 min to 11.000 min is subtracted as the background from spectrum averaged from retention time range of 10.000 min to 10.100 min.
[10.000 -> 10.100] - [10.500] - [10.040]	Records resultant spectrum where spectrum at 10.500 min retention time is subtracted from spectrum averaged from retention time range of 10.000 min to 10.100 min, and the spectrum at 10.040 min retention time is subtracted from it further.

Note

To save a resultant spectrum for future reference, register it in the Spectrum Process Table by selecting **Qualitative > Register to Spectrum Process Table**, or by right-clicking the spectrum and selecting **Register to Spectrum Process Table** from the pop-up menu. Select **Qualitative > Show Qualitative Table** to view the Spectrum Process Table. To display a spectrum saved in the table, select the appropriate row.

4.3 Peak Integration

This section describes how to integrate chromatogram peaks in the acquired data. The peaks can be integrated manually or automatically.

4.3.1 Manual Peak Integration

1. Click the **Manual Peak Integrate for TIC/MIC** toolbar button, or execute the **Qualitative > Manual Peak Integrate** command. 

2. When the cursor is moved over the chromatogram, it is displayed as a vertical line. Drag the cursor from the start of chromatogram peak that you wish to integrate to the end.

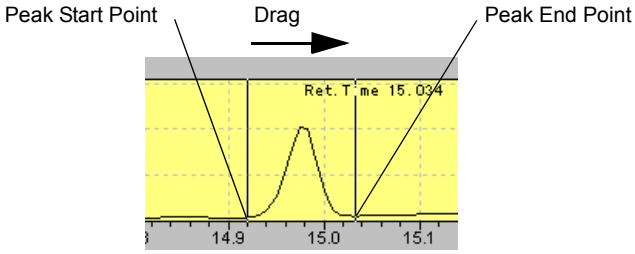


Figure 4.3 Peak Selection for Manual Integration

The "Select Base Line" dialog box is displayed.

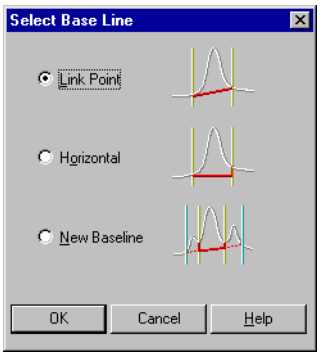
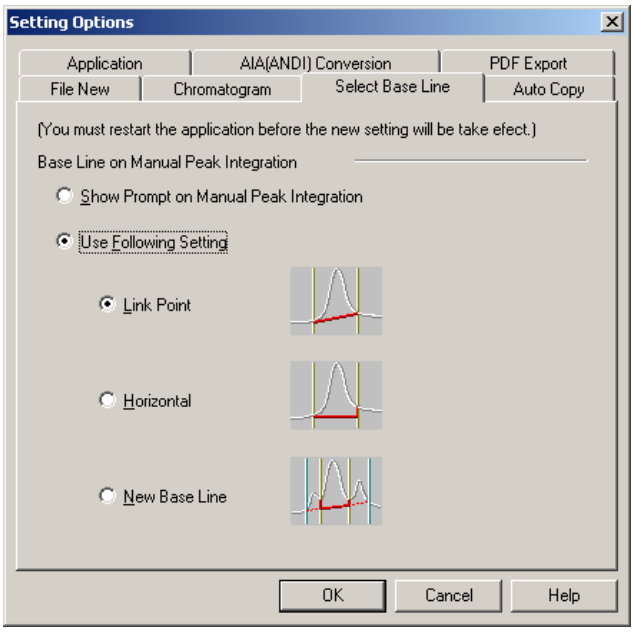


Figure 4.4 "Select Base Line" Dialog Box

Note

Select the **Tool > Option** command, the "Select Base Line" tab is displayed.



If Show Prompt on Manual Peak Integration button is selected, Select Base Line window is displayed when manually processing peaks. If Use Following Setting button is selected, the baseline is drawn as chosen in next three options without displaying the Select Base Line window.

3. Select how the baseline should be drawn from among Link Point, Horizontal, and New Baseline. Click the **OK** button.

- Link Point uses the beginning and ending time points selected on the chromatogram as the beginning and ending points for the baseline. The baseline beginning and ending points must overlap the chromatogram.
- Horizontal draws the baseline horizontally from the lowest intensity point between the selected chromatogram beginning and ending points. At least the beginning or ending point must overlap the chromatogram.
- After selecting **New Baseline** and clicking the **OK** button, use the drag function again to determine the baseline separately from the peak. The beginning and ending peak times are determined from the first drag. The baseline beginning and ending points are determined from the second, after closing the "Select Base Line" dialog box.

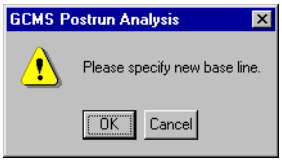


Figure 4.5 Specify a New Baseline

Note

After determining the baseline, the peak is marked on the chromatogram and the spectrum is displayed. If a spectrum for a different retention time is currently displayed, click the Peak buttons ( ) at the top of the Chromatogram View until the marked peak is selected to display its spectrum.

4.3.2 Automatic Peak Integration

1. Select **Qualitative > Qualitative Parameters**. The "Qualitative Parameters" dialog box is displayed.

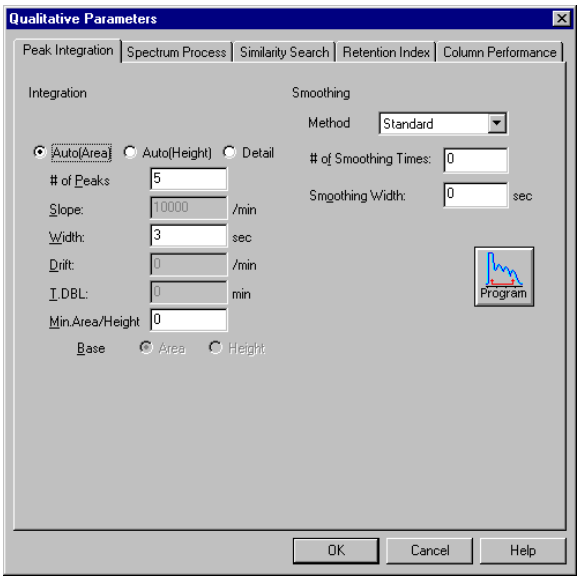


Figure 4.6 Qualitative Parameters Peak Integration Tab

2. Enter the parameters for Peak Integration in the Qualitative Parameters Peak Integration tab.

Peak Integration Parameters

Peak integration is performed as specified by the Peak Integration parameters. The **Program** button is displayed only when performing qualitative peak integration. The time program for quantitative peak integration is set from the Program cell in the Compound Table.

Parameter	Description	Units	Range
Integration	Selects how to integrate peaks from Auto (Area), Auto (Height), or Detail. Auto (Area): The slope is automatically adjusted until the entered # of Peaks is obtained. If the peak number is not obtained after adjusting the slope, the peaks with the largest areas are included to satisfy the peak number parameter. Confirm the final slope from the Qualitative Peak Table. Auto (Height): The slope is automatically adjusted until the entered # of Peaks is obtained. If the peak number is not obtained after adjusting the slope, the highest peaks are included to satisfy the peak number parameter. Confirm the final slope from the Qualitative Peak Table. Detail: Peak integration is based on the Slope, Width, Drift, and T.DBL parameters.		
# of Peaks	If Auto (Area) or Auto (Height) is selected as the integration method, this sets the number of peaks for it to target during slope adjustment. Depending on the chromatogram, this parameter may not be met directly. If the # of Peaks is not obtainable, the peaks with the largest areas or highest peaks are included until the parameter is satisfied. If Detail is selected as the integration method, this parameter is disabled.		
Slope	This parameter determines the sensitivity of peak detection. The slope, used to find the peak starting and end points, is calculated for tangents along the peak. The starting point of the peak is determined when the slope reaches a set value. The end point of the peak is determined when the negative slope reaches a set value. The starting and end points are calculated from a quarter value of the Width parameter. If the Slope parameter is high, the sensitivity will be low. If the Slope parameter is low, the sensitivity will be high, but broad peaks will be detected as well. Slope Parameter Precautions The Slope parameter changes at the time interval specified by the T.DBL parameter. To prevent the baseline from being detected as a peak, the slope must be greater than the drift for analyses with extreme baseline drift.	min^{-1}	0 - 4E+11

Parameter	Description	Units	Range
Drift	This parameter is used to determine the baseline. Normally, the value is set to 0, and the baseline is automatically corrected. If the automatic baseline correction is incorrect, enter a value that will obtain the proper baseline. The Drift should be larger than the slope from which the baseline is to be discerned. The Drift parameter leads to different results depending on whether the separation method provides complete or incomplete separation.	min^{-1}	–1E+7 - 1E+7
T.DBL	This parameter is the interval at which the Slope and Width are automatically changed. To prevent the Slope and Width from changing automatically, enter a value that is larger than the final analysis time. When the peak width increases with time, such as with GC isothermal analysis, the Slope and Width parameters must be changed over time. When the T.DBL is 0, the Slope and Width parameters change automatically as the peaks broaden. If the automatically determined interval is incorrect, enter a T.DBL parameter as specified by the formula below. The width is doubled and the slope is halved at each T.DBL interval. T.DBL (min) = (Width / Peak Width at Half-Height) x (Peak Retention Time) x 2 T.DBL Parameter Precautions The Width and Slope parameters change at a time interval determined by the T.DBL parameter.	min	0 - 10000
Width	This parameter is the width at half-height of the narrowest peak detected. The Width parameter is the standard used to distinguish between noise and peaks. As noise is narrower than peaks, if Width is set from the narrowest peak width at half-height, peaks that are narrower can be distinguished as noise and eliminated. If this value is not set correctly, errors in peak integration will occur. The Width parameter should be no greater than the width at half-height of the narrowest peak in the chromatogram. Width Parameter Precautions The Width parameter changes at the time interval specified by the T.DBL parameter. Automatic changes from the T.DBL parameter may occur up to 15 times per chromatogram, including the changes in a time program. For MS quantitative analysis, the width parameter can only change up to two times.	sec	0.04 - 200
Min. Area/ Height	Peaks with area/height less than this setting will not be recognized as peaks and will not be processed or output in reports.		
Base	This sets whether area or height will be used in Min. Area/Height. **Note:** The setting in the Quantitative Tab determine whether area or height is to be used in quantitative calculation.		

Parameter	Description	Units	Range
Smoothing	Select the Smoothing method from none, Standard, and Savitzky-Golay. None: Smoothing is not performed. Standard: Smoothing is performed from a moving average, as in the CLASS-5000 software. Savitzky-Golay: Smoothing is performed with the Savitzky-Golay method.		
# of Smoothing Times	If the Standard smoothing method is used, this parameter specifies how many times to perform smoothing. If the first moving average has little effect, set the # of Smoothing Times parameter. By smoothing multiple times, the moving average effects are enhanced. Smoothing is not performed when the # of Smoothing Times is 0. This parameter is disabled if None or Savitzky-Golay is selected as the smoothing method.		0 - 99
Smoothing Width	If the Standard smoothing method is used, this parameter specifies the average width for the moving averaging in seconds. If the Savitzky-Golay smoothing method is used, this parameter specifies the number of points to smooth. Only odd numbers of points are effective. Even if an even number is entered, an odd number of points is smoothed. This parameter is disabled if None is selected as the smoothing method.	sec Points	0 - 200 3 - 25

Note

Refer to Appendix A "Peak Processing and Mass Spectrum Operations" on page 273, Section A.1 "Integration and Peak Processing Parameters", page 273 and Section A.5 "Troubleshooting", page 304 for more information.

Time Program for Peak Integration

After clicking the **Program** button, the "Time Program for Peak Integration" dialog box is displayed.

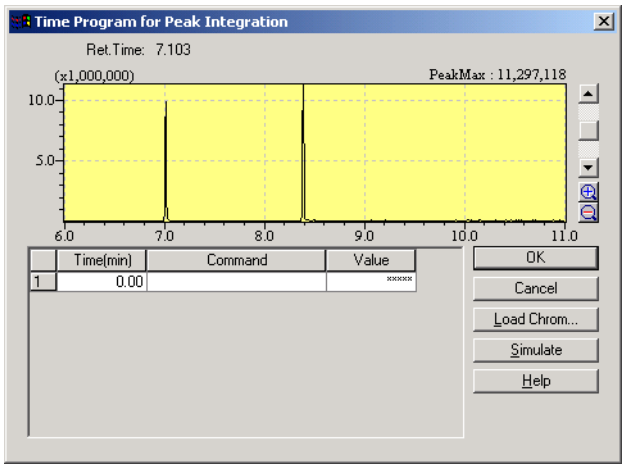


Figure 4.7 "Time Program for Peak Integration" Dialog Box

If optimal peak processing cannot be obtained with the default method parameters, use this dialog box to create a time program to change peak integration parameters over time.

When peak integration is initiated, qualitative analysis proceeds with the parameters entered in the **Peak Integration tab**. The time program changes the parameters at set times. Peak processing continues with the changed parameters.
Use a previously acquired chromatogram to facilitate time program development. Select the **Load Chrom** button, then choose the appropriate data file.

Click in the Time column, then click the chromatogram at the desired time. The retention time is automatically entered into the table. Select a command from the drop down list, then enter numerical values for commands that are followed by an "=".

Note

For FASST measurement interval, the Scan TIC is displayed.

Command	Description	Units	Range
INTEGRATION ON/OFF	Peaks that occur between INTEGRATION OFF and ON are considered unnecessary, and are not integrated.		ON/OFF
TAILING ON/ OFF/AUTO	This program performs tailing processing automatically. Forced tailing processing is conducted between the TAILING ON and TAILING OFF times specified. For TAILING AUTO, tailing processing is performed automatically from the time specified.		ON/OFF/AUTO

Command	Description	Units	Range
HORIZ BASELIN ON/ OFF	The baseline of peaks within the HORIZ BASELIN ON to HORIZ BASELIN OFF interval is processed to become horizontal.		ON/OFF
PEAK DETECT/TOP/ END	Two or more peaks are forced to be processed as one peak.		DETECT/TOP/ END
LEADING PK ON/OFF	Leading (fronting) processing is conducted within the LEADING PK ON to LEADING PK OFF interval.		ON/OFF
WIDTH=	Changes the Width peak integration parameter. Precautions The Width value changes over time when T.DBL is used. For further information, refer to the T.DBL parameter in the Peak Integration Parameters table. Normally, the Width command should only be used in a time program when the T.DBL command is disabled (the T.DBL value is larger than the analysis ending time). The use of a valid T.DBL value combined with a WIDTH = command is treated by the program in the following way: When a user-specified value has been set for T.DBL: Initially, the WIDTH value is equal to the Width value at analysis start. The current Width value is calculated with T.DBL. Then the Width value is doubled each time the T.DBL time is reached. When T.DBL is set to 0 (automatic T.DBL): The Width value is changed at the time set in the program. Then the Width value is doubled according to the width of the peak. Limit of width changes per analysis (including analysis with T.DBL): up to 15 times. For MS Quantitative processing: up to 2 times.	sec.	0.04 - 200
SLOPE=	Changes the Slope peak integration parameter. Precautions The Slope value changes with time when using T.DBL. For further information, refer to the T.DBL parameter in the Peak Integration Parameters table. Normally, the Slope command should only be used in a time program when the T.DBL command is disabled (the T.DBL value is larger than the analysis ending time). The use of a valid T.DBL value combined with a SLOPE= command is treated by the program in the following way: When a user-specified value has been set for T.DBL: Initially, the SLOPE parameter is equal to the Slope value at analysis start. The current Slope value is calculated with T.DBL. Then the Slope value is halved each time the T.DBL time is reached. When T.DBL is set to 0 (automatic T.DBL): The Slope value is changed at the set time in the program. Then the Slope value is halved according to the width of the peak.	min^{-1}	0.0 - 4E+11
DRIFT=	Changes the Drift peak integration parameter.	min^{-1}	–1E+7 - 1E+7

Command	Description	Units	Range
T.DBL=	Sets the T.DBL peak integration parameter. Precautions T.DBL changes the values for the Slope and Width peak integration parameters over time. Normally, do not combine this command with a WIDTH or SLOPE command that changes Slope or Width parameters in a time program. Processing with a user-specified T.DBL: Initially, the T.DBL parameter is equal to the T.DBL value at analysis start; the current Width and Slope values are calculated with this value. Then the Width and Slope values are changed (Width doubled, slope halved) at the specified time. Auto Processing (T.DBL setting 0): The Slope and Width peak processing parameter values are changed from their starting values; T.DBL processing is conducted automatically.	min.	0.0 - 10000.0

Note

Refer to Appendix A "Peak Processing and Mass Spectrum Operations" on page 273, Section A.1 "Integration and Peak Processing Parameters", page 273 and Section A.5 "Troubleshooting", page 304 for more information.

3. After entering each parameter in the Peak Integration table, click the **OK** button. Peak marks and baselines are added to the chromatogram displayed in the Chromatogram View, and the peaks are integrated.

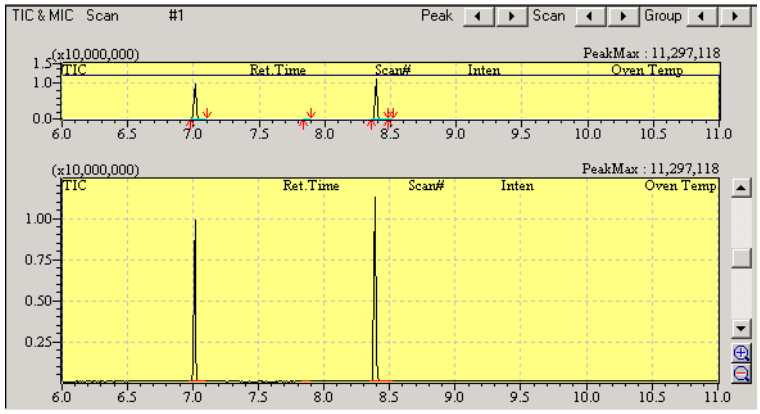


Figure 4.8 Chromatogram View

4.3.3 Manipulation

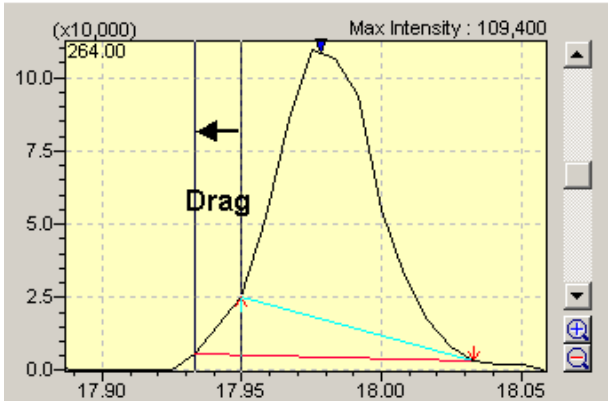
There are Move the Detection Point and Split Peak in manipulation. These can be performed in the Chromatogram view, Quantitative view in Data Analysis window, Single/ Multi Chromatogram Display view in Calibration window or Chromatogram view (Single) in Quantitative Browser window.

1. Move the Detection Point
This allows the cursor to be moved to specify the start point and the end point of peak.

Note

Tailing or leading edges of that peak can be moved. The peak detection points cannot be moved into the region of adjacent peaks.

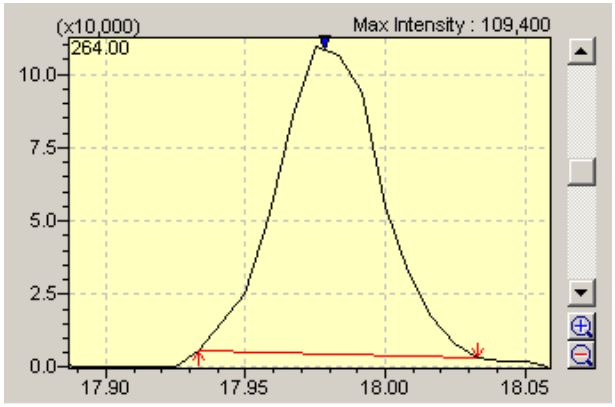
(1) If the mouse pointer is moved near a detecting point, the cursor changes to triangle.
(2) Drag the cursor from the previous detecting point to the new detecting point. While the mouse cursor is moving, the cursor changes to a vertical line.



Note

While the mouse cursor is moving, the drawn baseline (red line) changes according to the cursor position.

(3) When the detecting point is moved to the new position, data processing is performed, and Mark, Baseline, Area, Height and Conc. are updated.



Note

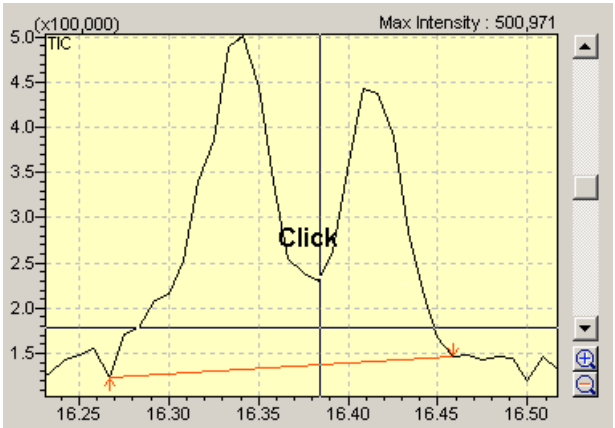
The mark of peak processing is expressed as "MI" for the manual peak processing.

2. Split Peak

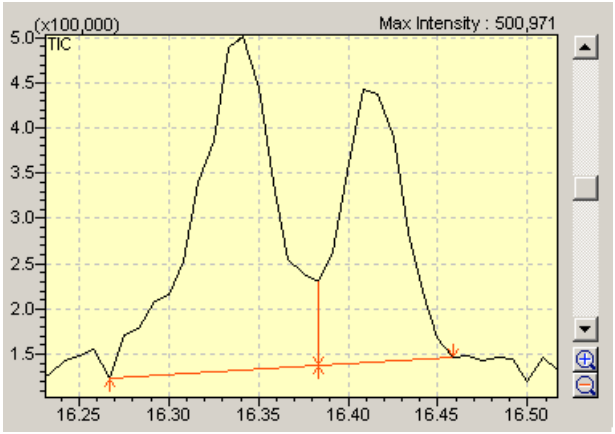
If this command is selected, the chromatogram area will become the Split Peak mode, and a peak can be divided at the specified retention time. In order to cancel this mode, please select this command again.

(1) When the Split Peak command is selected from the Qualitative menu, the mouse pointer on the chromatogram changes to a cross line.

(2) Click at the splitting point of the peaks. The boundary line is drawn vertically at the point.



Peak processing is performed for both peaks. Mark, Baseline, Area, Height and Conc. are updated for each peak.



Note

The mark of peak processing is expressed as "MI" for the manual peak processing.

4.3.4 Peak Integration by Batch Processing

Refer to Chapter 7 "Continuous Analysis" on page 175 for information about integrating peaks during batch processing.

4.4 Similarity Search

This section describes how a spectrum shown in Spectrum View can be compared to similar spectra in a library file. It also describes the comparison between candidate and target spectra, and the identification of components.

4.4.1 Similarity Search for Displayed Spectrum

1. Click the Peak buttons ( ) at the top of the Chromatogram View to select a peak to identify with the similarity search. The spectrum of the selected peak, or target spectrum, is displayed in the Spectrum View.

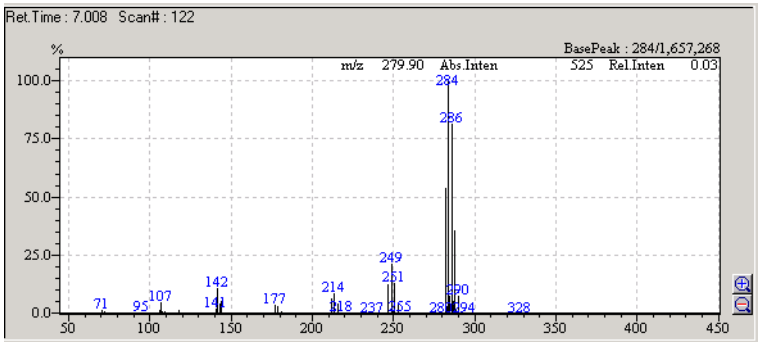


Figure 4.9 Spectrum View

2. Click the **Similarity Search Result** toolbar button to open the "Similarity Search Results" window.

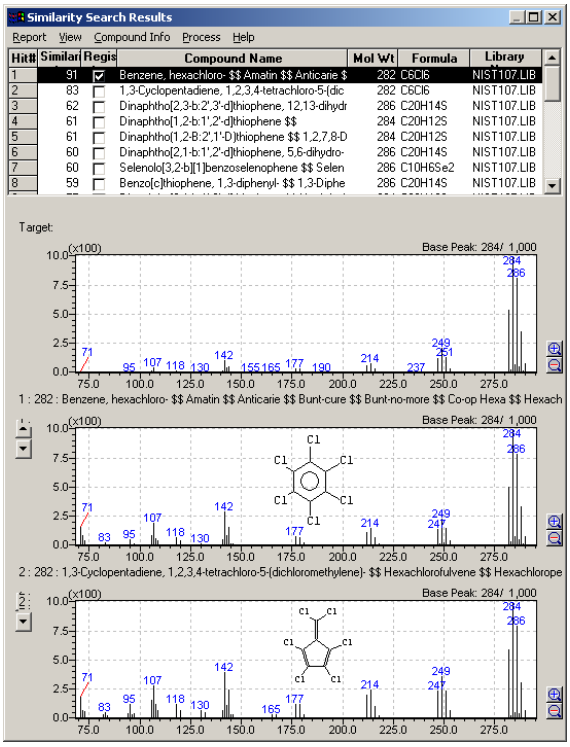


Figure 4.10 "Similarity Search Results" Window

Compounds are listed in the "Similarity Search Results" window sequentially, starting with compounds having the highest degree of similarity. The list can be scrolled from below with the ( ) buttons to the left of the middle spectrum. The middle spectrum changes to the spectrum of the compound that is currently selected in the list.

The top spectrum is the target spectrum, and the second spectrum is a candidate compound spectrum from the library. The information in the lower portion of the window varies depending on which display option is currently selected in the View menu. If Compare is selected, the lower portion contains a second candidate spectrum from the same library as the second spectrum. If Information is selected, the compound information about the candidate spectrum is displayed. If Subtract is selected, the lower portion displays the resultant spectrum from subtracting the candidate spectrum from the target spectrum.

Note

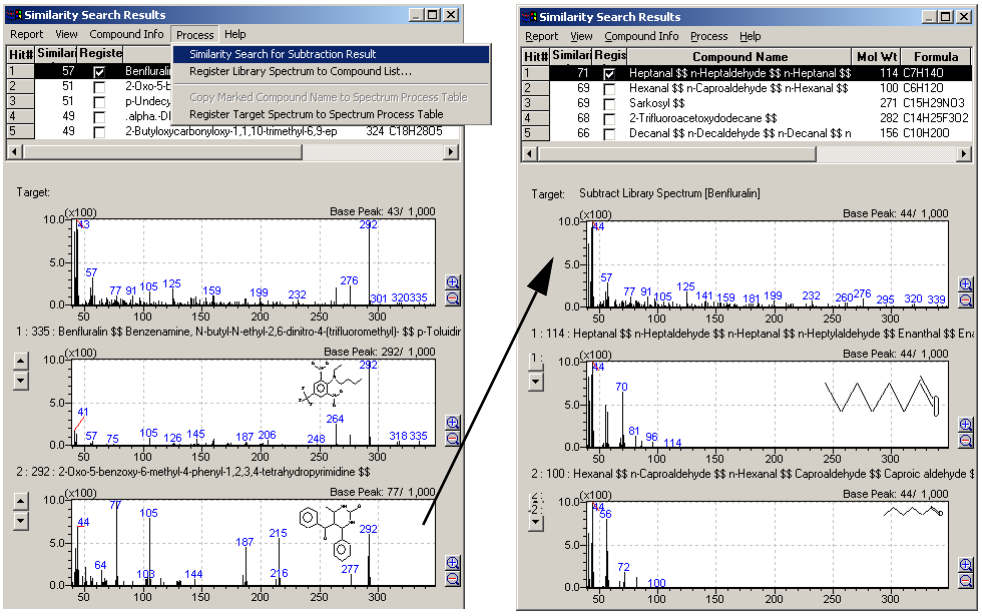
The library file used for the Similarity Search is selected in the Similarity Search tab of the "Qualitative Parameters" dialog box. To open the "Qualitative Parameters" dialog box, either click the **Qualitative Parameters** toolbar button or select **Qualitative > Qualitative Parameters**.
Five library files can be selected under Library File Name. A minimum similarity index may be entered for each file under Min.SI. The maximum number of hits and other parameters are also specified in this tab. Refer to GCMS Help for more information about the Similarity Search parameters.

4.4.2 Similarity Search for Subtraction Spectrum Repeatedly

When you know previously that the target spectrum consists of spectrum of multiple compounds, by subtracting the spectrum hit by similarity search and executing similarity search again, you can obtain a more accurate search result.

1. Select the **Process > Similarity Search for Subtraction Result** command.

2. A new Similarity Search window opens, and the subtracted spectrum and the search results are displayed.



3. You can also execute similarity search for the subtraction result again. (up to 2 times consecutively)

4.4.3 Specify Library Spectrum

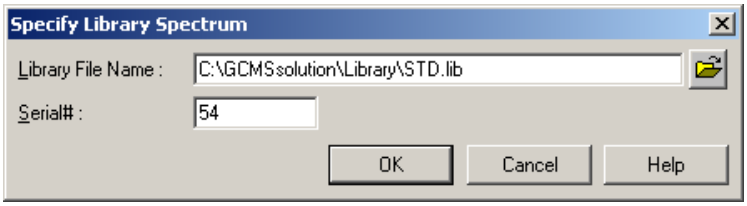
You can add the library spectrum to the end of the hit list on Similarity Search window.

By doing this, you can display the similarity search results of the spectrum and the similarity index, then also can execute Similarity Search for Subtraction Result.

1. Select the **Process > Register Library Spectrum to Compound List** command.

2. The "Specify Library Spectrum" window is opened.
   Enter the library file name and serial number of spectrum which you want to add to the Hit List.

.



4.5 Printing Results

This section explains how to print the default qualitative analysis reports.

4.5.1 Chromatogram and Spectrum

To print a report of the chromatogram and spectrum displayed during qualitative analysis, select **File > Print Image > Print** or click the **Print** toolbar button. Select **File > Print Image > Preview**, or click the **Print Preview** toolbar button to view the report on-screen before printing. A default report format is used to print the chromatogram and spectrum. To edit the default Image report format, select **File > Print Image > Edit Format**.

Print Image Report

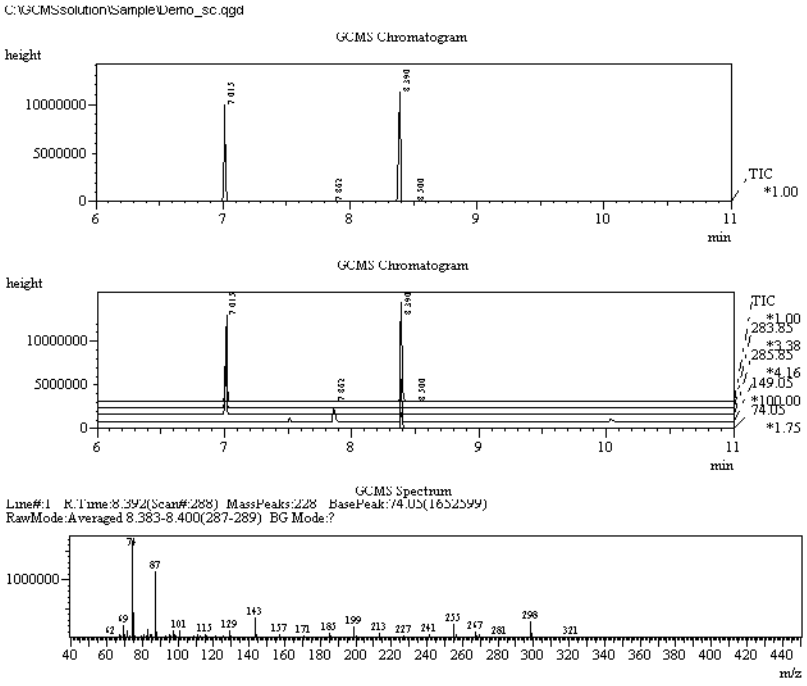


Figure 4.11 Printed Report

4.5.2 Similarity Search Results

Use the Report menu in the "Similarity Search Results" window to print the three default reports described below.

Menu command	Description
Search Results	Prints the target spectrum and the currently displayed candidate compound spectrum.

Menu command	Description
Comparison	Prints the target spectrum, the currently displayed candidate compound spectrum, and the resultant spectrum from subtracting the candidate spectrum from the target spectrum.
Cmpd Info	Prints the compound information, mass table, structural formula, and other data about the candidate compound from the library.

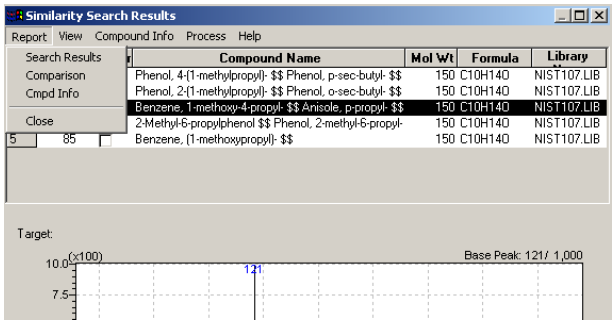


Figure 4.12 Similarity Search Results Report Selection

4.5.3 Custom Report

To print a custom report from an open data file, select **File > Report**. The "Report" window is displayed. Either create a new report format and select the currently opened data file as the data file for each item, or open an existing report format file and select the currently opened data file as the data file for each item. Print the report. Refer to Chapter 6 "Generating Custom Reports" on page 165 for information about creating custom report formats.

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5 Quantitative Analysis

5.1 Overview

This section describes processing the acquired data with the GCMS Postrun Analysis application. It also explains compound identification, calibration curve generation, and concentration calculations, and printing of reports.

5.1.1 "Postrun Analysis" Windows

1. The "Data Analysis" Window

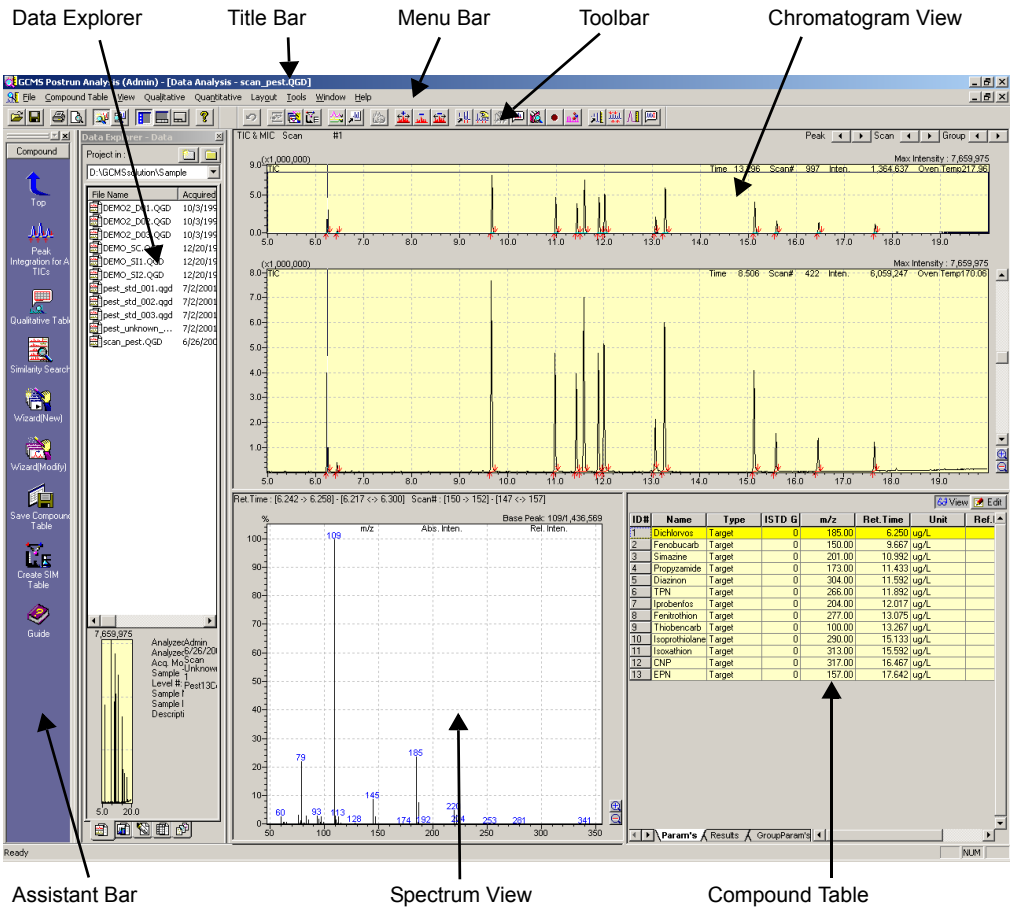


Figure 5.1 The "Data Analysis" Window

The items in this screen are described in the following table.

Title Bar	Displays the name of the application, user, active window, and file.
Menu Bar	Displays the menus that are available in the active window.
Toolbar	Displays the command buttons that are available in the active window.
Assistant Bar	Displays the command icons that apply to a specific function or window. To open the window or activate the command, click the appropriate icon.
Data Explorer	Displays all files by type, including Data, Method, Report Format, Batch, and All Files. Data can easily be opened for analysis by double-clicking the file icon or dragging it to the appropriate location.
Chromatogram View	Displays the chromatogram from the open data file.
Spectrum View	Displays the mass spectrum for a retention time or compound selected in the Compound Table.
Compound Table	Displays the following tables: • Compound table: Contains parameters for compound identification, calibration curve creation, and concentration calculations. • Compound Result table: Displays the results of compound identification and concentration calculations. • Grouping table: Contains parameters for calibration curve creation and concentration calculations of the group. • Grouping Result table: Displays the results of the grouping depending on the parameters on the Grouping table.

2. The "Calibration Curve" Window

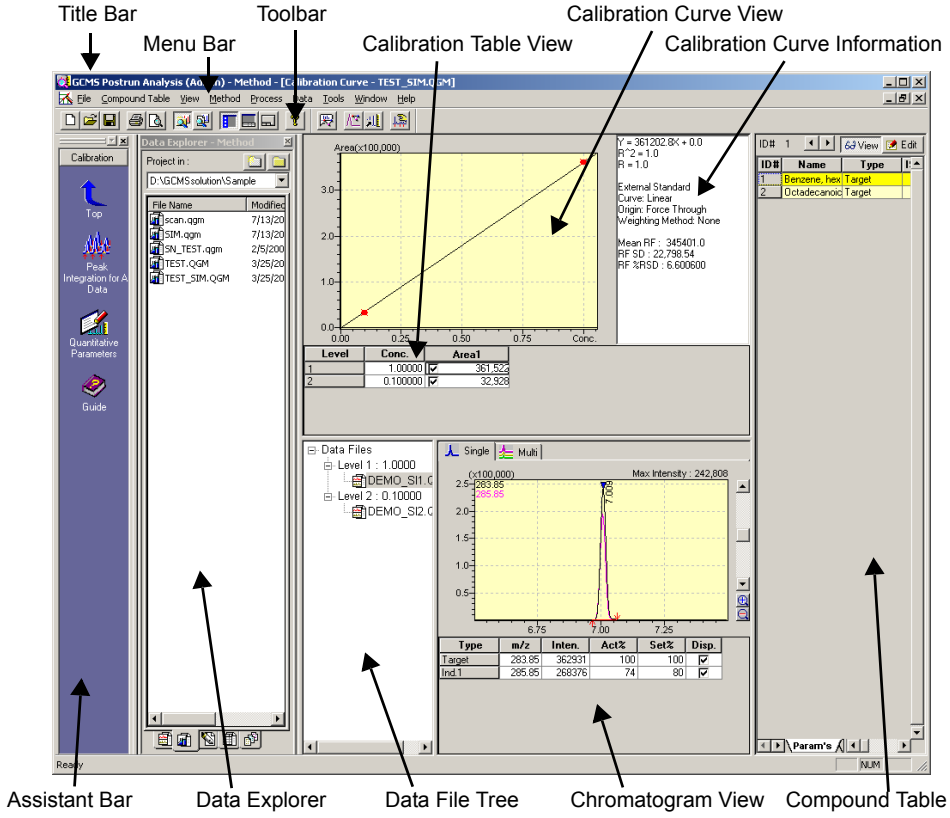


Figure 5.2 The "Calibration Curve" Window

Title Bar	Displays the name of the application, user, active window, and file.
Menu Bar	Displays the menus that are available in the active window.
Toolbar	Displays the command buttons that are available in the active window.
Assistant Bar	Displays the command icons that apply to a specific function or window. To open the window or activate the command, click the appropriate icon.
Data Explorer	Displays all files by type, including Data, Method, Report Format, Batch, and All Files. Method files can easily be opened for calibration curve creation by double-clicking the file icon and data files can be dragged to the Data File Tree for analysis.
Calibration Curve View	Displays the calibration curve for a compound selected in the compound or results table.
Calibration Curve Information	Displays the type of calibration curve displayed and results of statistical calculations.
Calibration Table View	Displays the concentration, and response factor or area/height of each level in the currently displayed calibration curve.
Data File Tree	Displays the data files that can be accessed to create the calibration curve at each level. The selected data file is the one to which the currently displayed compound and quantitative results tables belong.
Chromatogram View	Displays the chromatogram for a compound selected in the compound or quantitative results table.
Compound Table	Displays the following tables: • Compound table: Contains parameters for compound identification, calibration curve creation, and concentration calculations. • Compound Result table: Displays the results of compound identification and concentration calculations. • Grouping table: Contains parameters for calibration curve creation and concentration calculations of the group. • Grouping Result table: Displays the results of the grouping depending on the parameters on the Grouping table.

3. "Quantitative Data Analysis" Window

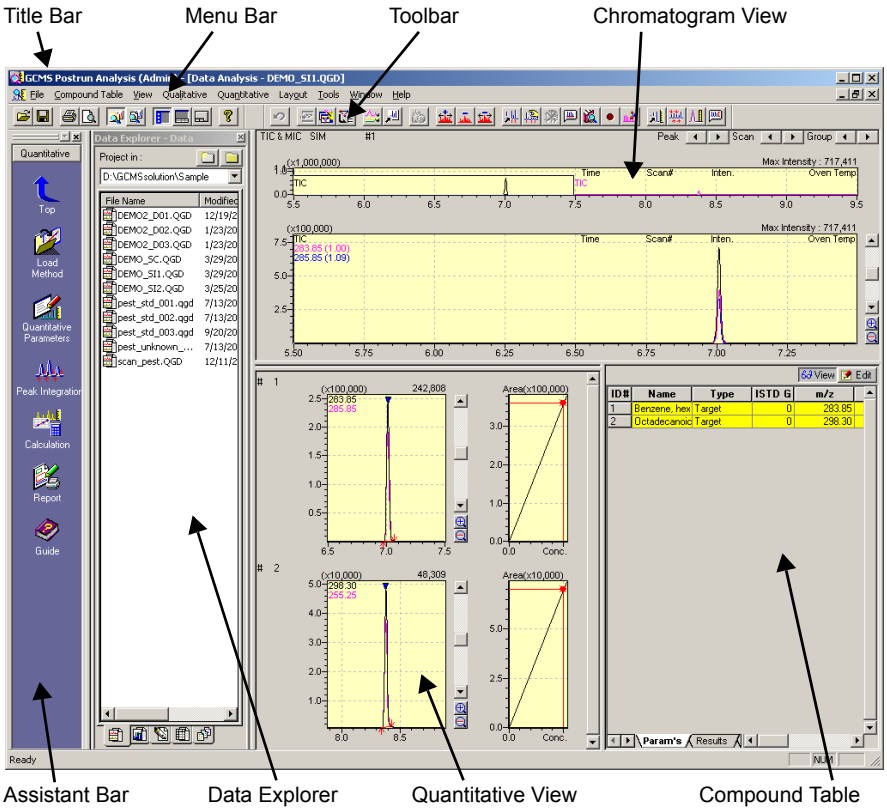


Figure 5.3 "Quantitative Data Analysis" Window

Title Bar	Displays the name of the application, user, active window, and file.
Menu Bar	Displays the menus that are available in the active window.
Toolbar	Displays the command buttons that are available in the active window.
Assistant Bar	Displays the command icons that apply to a specific function or window. To open the window or activate the command, click the appropriate icon.
Data Explorer	Displays all files by type, including Data, Method, Report Format, Batch, and All Files. Data can easily be opened for analysis by double-clicking the file icon or dragging it to the appropriate location.
Chromatogram View	Displays the chromatogram from the opened data file.
Quantitative View	Displays the calibration curve and chromatogram for the compound selected in the compound or quantitative results table. Displays data for up to five compounds at a time.
Compound Table	Displays the following tables: • Compound table: Contains parameters for compound identification, calibration curve creation, and concentration calculations. • Compound Result table: Displays the results of compound identification and concentration calculations. • Grouping table: Contains parameters for calibration curve creation and concentration calculations of the group. • Grouping Result table: Displays the results of the grouping depending on the parameters on the Grouping table.

5.1.2 Quantitative Analysis Procedures

The procedures for quantitative analysis are described below.

Method Development	The method file contains the instrument parameters for the GC and MS and the qualitative, quantitative, data view, and QA/QC parameters. Create a new method file with GCMS Real Time Analysis, or use an existing method file after verifying its contents. For more information about method development, see Section 3.2 "Method Development Parameters", page 90.
↓	
Standard Injection and Data Acquisition	Data is acquired in GCMS Real Time Analysis. Enter information about single standards in the "Data Acquisition" window, or enter information about multiple standards in the "Batch Table" window. Initiate single or batch processing. For more information about data acquisition, refer to Section 3.3 "Single Run Setup", page 104 and Section 3.4 "Data Acquisition", page 107.
↓	
Compound Table Creation	Open GCMS Postrun Analysis, and click the Assistant Bar **Quantitative Analysis** icon. Open the newly acquired data file(s), and analyze the data. Identify the standard compounds, and create the compound table as described in this chapter. Refer to Section 5.2 "Compound Table", page 143.
↓	
Calibration Curve Generation	Generate the calibration curve from the compound table. Refer to Section 5.3 "Creating Calibration Curves", page 149.
↓	
Unknown Sample Injection and Data Acquisition	Acquire data for unknown samples with GCMS Real Time Analysis, and perform concentration calculations. Refer to Chapter 3 "Data Acquisition", page 85.
↓	
Quantitative Calculations and Verification of Quantitative Results	Identify compounds through quantitative calculations, and confirm the concentration calculation results. Refer to Chapter 5.4 "Performing Quantitation and Confirming Results", page 151.
↓	
Similarity Search	Compounds in the library with spectra that are highly similar to the spectrum of the identified compound in the compound table are listed sequentially from the most similar, and the spectra are compared. Refer to Chapter 5.5 "Similarity Search", page 155.

The Quantitative Analysis Process

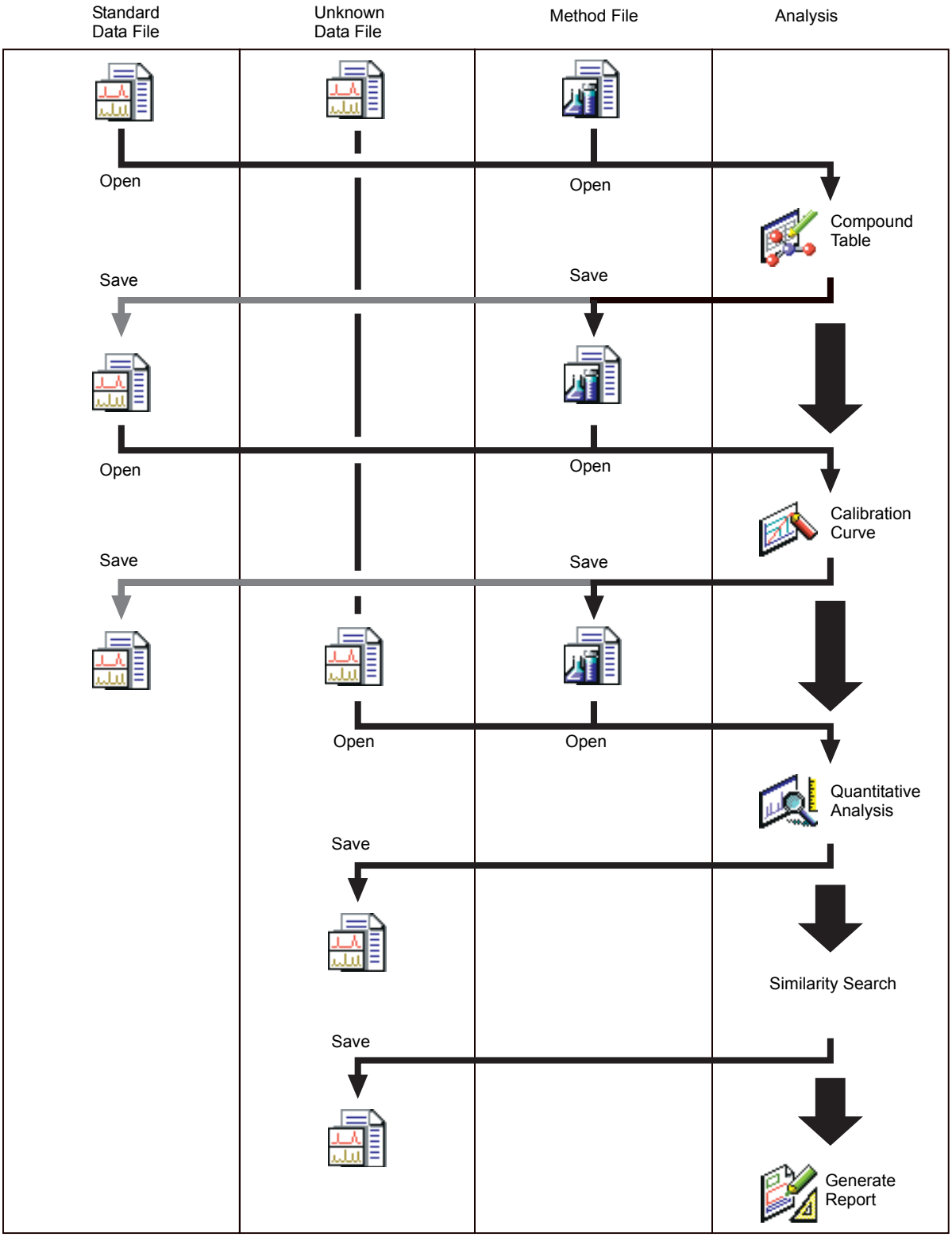


Figure 5.4 The Quantitative Analysis Process

5.2 Compound Table

This section explains how to create the compound tables that are necessary for peak integration, quantitation, and calibration curve generation. The information may be entered into the Compound Table directly, or the Compound Table Wizard may be used to facilitate the creation process.

1. Open acquired data.
   Start GCMS Postrun Analysis, and click the Assistant Bar **Create Compound Table** icon. The "Data Analysis" window is displayed in the compound table mode.

2. Click the Data Explorer Data tab, and double-click the data file icon for the standard. The data is displayed in the Chromatogram View.

3. Click the Assistant Bar **Wizard (New)** icon. The compound table enters the edit mode, and the Compound Table Wizard is displayed.

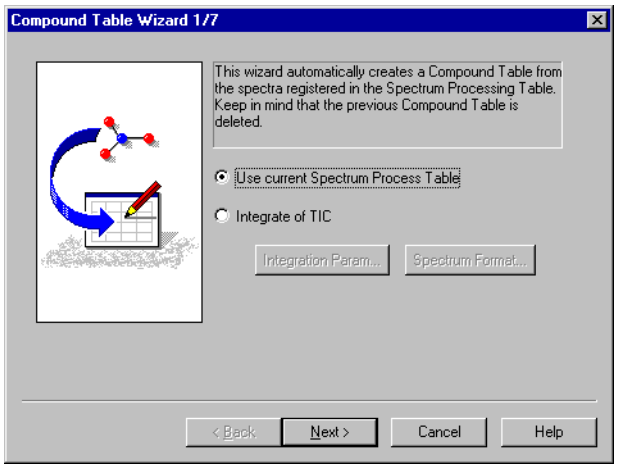
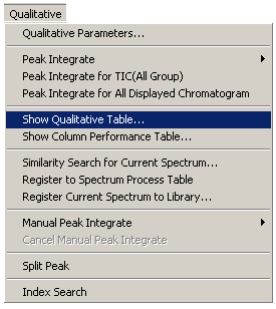


Figure 5.5 Compound Table Wizard Screen 1/7

4. Complete the Compound Table Wizard 1/7 screen.

   Select the **Use Current Spectrum Process Table** or **Integrate of TIC** radio button.

   To check the contents of the Spectrum Process Table before selecting Use Current Spectrum Process Table, click the **Cancel** button and temporarily close the Wizard. Select **Qualitative > Show Qualitative Table** or click the Assistant Bar **Qualitative Table** icon to open the "Qualitative Table" window. Verify the Spectrum Process Table on the Spectrum Process tab. Click the Assistant Bar **Wizard (New)** icon to reopen the Compound Table Wizard.



Note

Refer to GCMS Help for more information about the "Qualitative Table" window.

When "Integrate of TIC" is selected, the **Integration Param** and **Spectrum Format** buttons are enabled. Click the **Integration Param** button to verify the contents of the Peak Integration tab of the "Qualitative Parameters" dialog box. Click the **Spectrum Format** button to verify the contents of the Spectrum Process tab of the "Qualitative Parameters" dialog box.

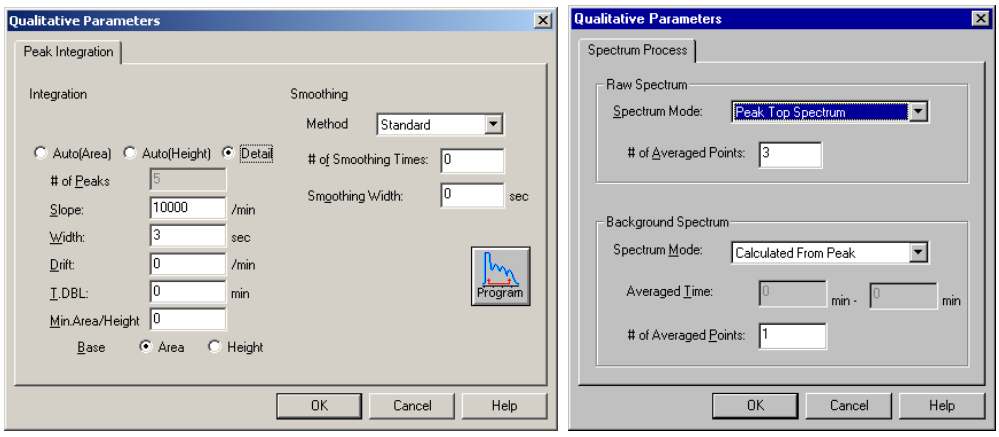


Figure 5.6 Peak Integration and Spectrum Format Parameters

Note

Refer to GCMS Help for more information about setting the parameters in the Peak Integration and Spectrum Process tabs.

Click the **Next** button to proceed to the Compound Table Wizard 2/7 screen.

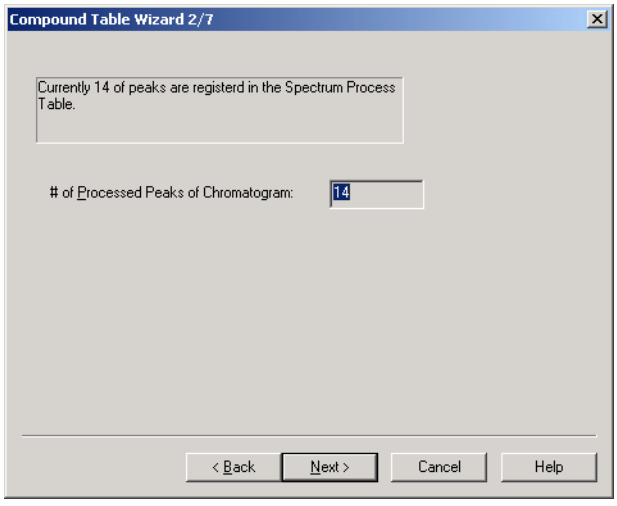


Figure 5.7 Compound Table Wizard 2/7 Screen

5. Complete the Compound Table Wizard 2/7 screen.
The number of peaks registered in the current spectrum processing table is displayed as the value for this text box.

Click the **Next** button to proceed to the Compound Table Wizard 3/7 screen.

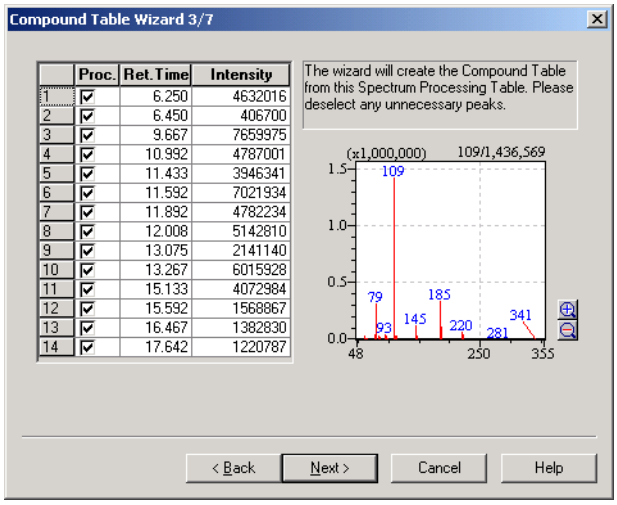


Figure 5.8 Compound Table Wizard 3/7 Screen

6. Complete the Compound Table Wizard 3/7 screen.

The Compound Table is constructed from the peaks in the spectrum process table on the screen that are checked in the Proc. column. If peaks that should not be included in the Compound Table are checked, click the check box to uncheck them. Highlighting a row displays the mass spectrum for the selected compound.

Click the **Next** button to proceed to the Compound Table Wizard 4/7 screen.

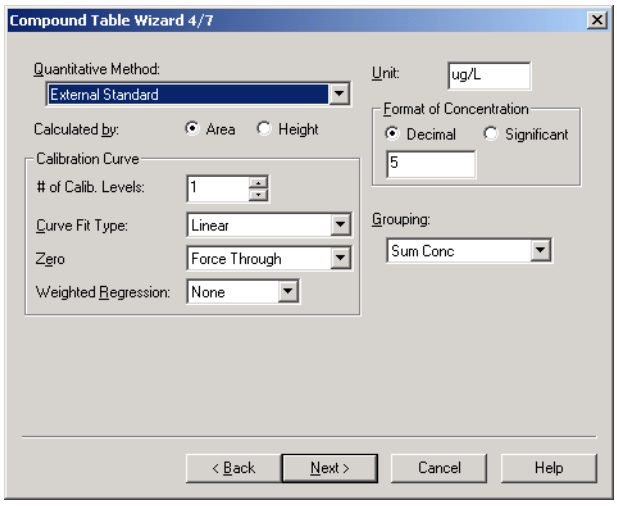


Figure 5.9 Compound Table Wizard 4/7 Screen

7. Complete the Compound Table Wizard 4/7 screen.
Enter the quantitative method, calibration curve, and concentration parameters.

Note

Refer to GCMS Help for more information about setting the Compound Table Wizard 4/7 screen parameters.

Click the **Next** button to proceed to the Compound Table Wizard 5/7 screen.

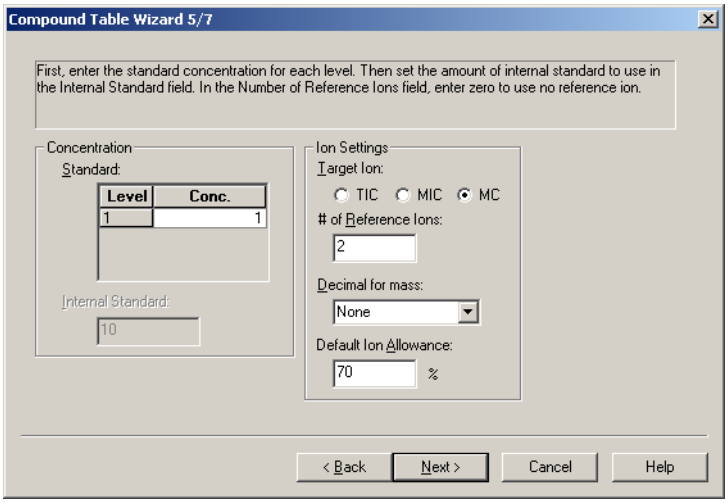


Figure 5.10 Compound Table Wizard 5/7 Screen

8. Complete the Compound Table Wizard 5/7 screen.

Enter the concentration for each level in the calibration curve, the number of reference ions to use in peak identification, and a decimal format for masses. If Internal Standard is selected as the Quantitative Method in Compound Table Wizard 4/7, enter an Internal Standard amount as well.

Click the **Next** button to proceed to the Compound Table Wizard 6/7 screen.

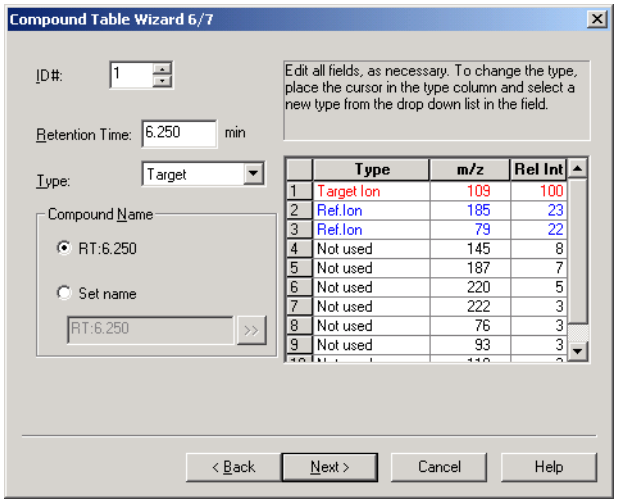


Figure 5.11 Compound Table Wizard 6/7 Screen

9. Complete the Compound Wizard 6/7 screen.

Edit the information for each compound ID. To change compounds, enter an ID number directly or use the spin control arrows to the right of the ID# text box. The spectrum corresponding with the selected ID is displayed in the spectrum view.

Select either Reference or Target for the compound type in the Type combo box.

Designate a compound name. When "Use the Current Spectrum Processing Table" is selected in the Compound Table Wizard 1/7 and the compound name is designated in the table, that compound name is displayed. The first radio option depends upon how the compound was entered in the Spectrum Process Table. If a Similarity Search was performed on the Spectrum Process Table, the name of the first hit compound in the library is the first name option. If a Similarity Search has not been performed, the first name option is "RT:" followed by the retention time. When the Set Name option is selected, a name may be entered into the text box.

In the table on the right side of the screen, the Target Ion and Reference Ion are listed sequentially from the ions having largest relative intensity.

To change a cell in the Type column, click the cell. The cell becomes a combo box; select Target Ion, Reference Ion, or Not Used. If changing the target or reference ion, first change the Target Ion or Reference Ion to Not Used, then change the other ion to Target Ion or Reference Ion.

To change a m/z, click the appropriate cell. Click the button which appears in the cell, and the spectrum is displayed. Determine the position of the spectrum from which to get the m/z, and double-click that location. The m/z is selected, and the reference ion ratio is recalculated.

Click the **Next** button to proceed to the Compound Table Wizard 7/7 screen.

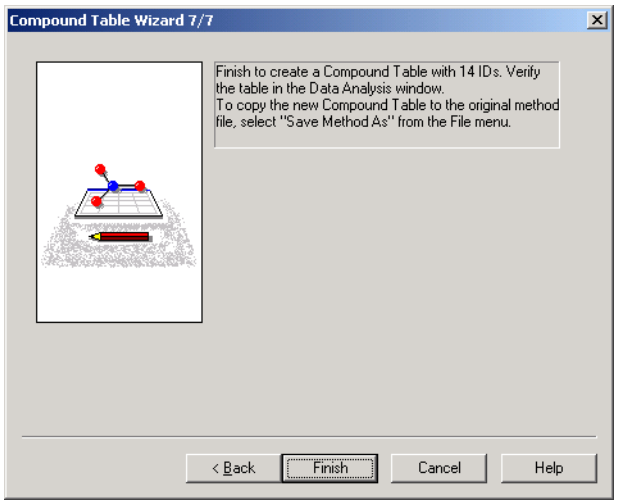


Figure 5.12 Compound Table Wizard 7/7 Screen

10. Complete the Compound Table Wizard 7/7 screen.

After confirming that a compound table with the required number of compounds, as designated in the Compound Table Wizard 3/7 screen, will be created, click the **Finish** button. The Compound Table will be displayed in the Param's tab of the Compound Table View.

11. Set the quantitative parameters.

After creating the compound table, modify the quantitative parameters as necessary. Select **Quantitative > Quantitative Parameters** or click the **Quantitative Parameters** toolbar button. The "Quantitative Parameters" dialog box is displayed.

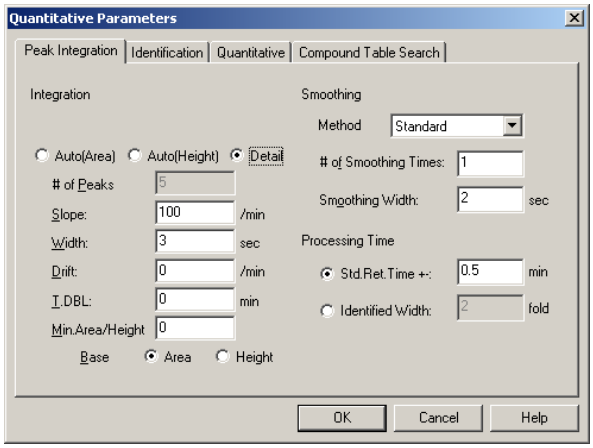


Figure 5.13 "Quantitative Parameters" Dialog Box

After verifying that the parameters are correct, save the method and data files.

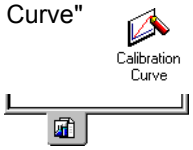
Note

The Qualitative Parameters time program is accessed from the Peak Integration tab. For Quantitative Parameters, the Time Program for Peak Integration is accessed from the Program column in the Compound Table.

5.3 Creating Calibration Curves

This section explains how to create a calibration curve with the data in the compound table.

1. Click the Assistant Bar **Calibration Curve** icon, and the "Calibration Curve" window is displayed.



2. Click the Method tab of the Data Explorer, and double-click the method file from which to create the calibration curve. The compound table saved with the method file is displayed.

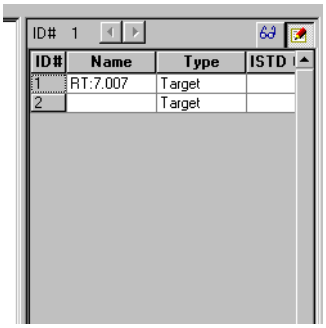


Figure 5.14 Compound Table

3. Click the Data tab of the Data Explorer, select the data file from which to create the calibration curve, and drag it from the Data Explorer to the Data File Tree.

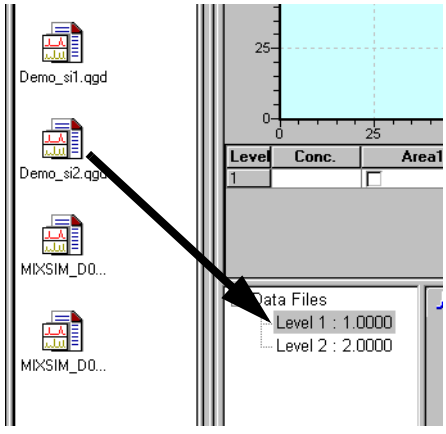


Figure 5.15 Drag the Data File Icon to the Data File Tree

Note

When creating a calibration curve with two or more levels, drag the data files for each level to its corresponding location in the data file tree.

4. Click the Assistant Bar **Peak Integration for All Data** icon.
The calibration curve is generated and displayed in the Calibration Curve View.

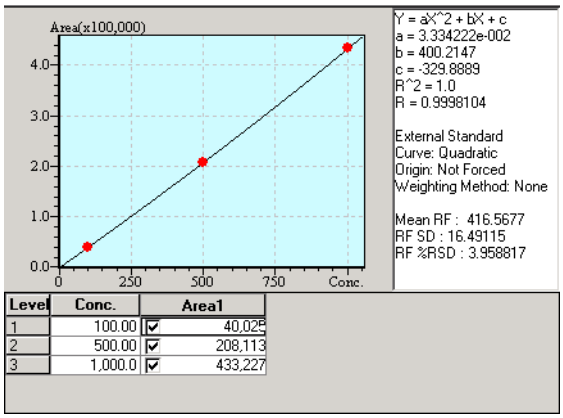


Figure 5.16 Calibration Curve View

Save the method file.

5.4 Performing Quantitation and Confirming Results

This section explains how to perform quantitative processing calculations and view the results. Compound tables and calibration curves are discussed as well.

1. Click the Assistant Bar **Quantitative** icon, and the "Quantitative Data Analysis" window is displayed.

2. Open the data file on which to perform quantitation.

   Click the Data tab in the Data Explorer, and double-click the data file icon to open the file. The data is displayed in the respective Views.

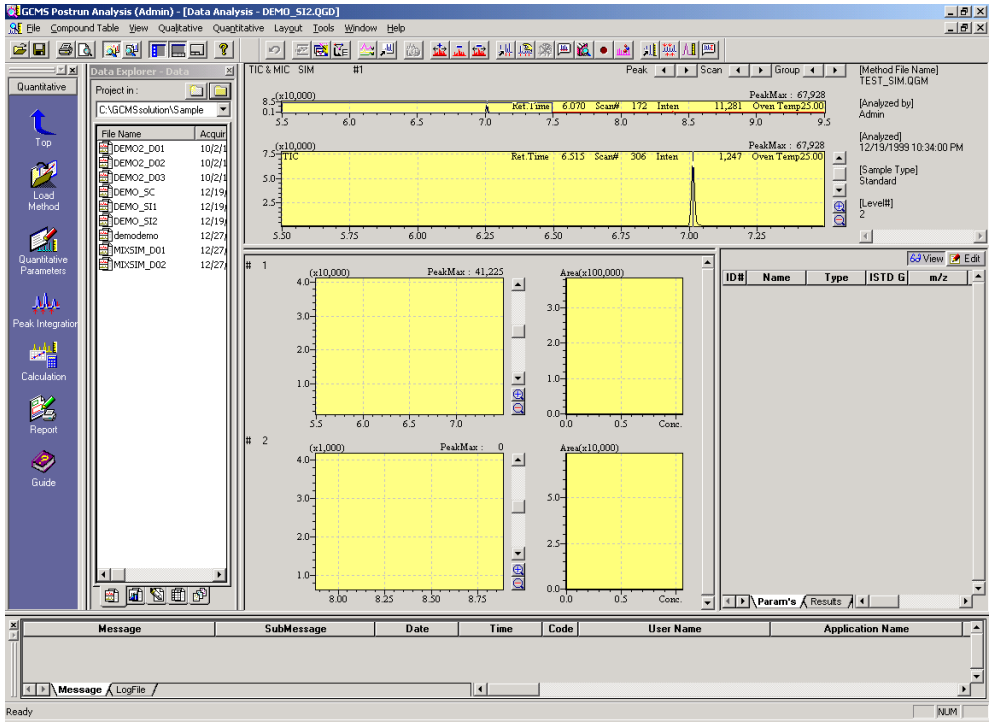


Figure 5.17 "Quantitative Data Analysis" Window

3. Click the Assistant Bar **Load Method** icon, and the "Load Method" dialog box is displayed.

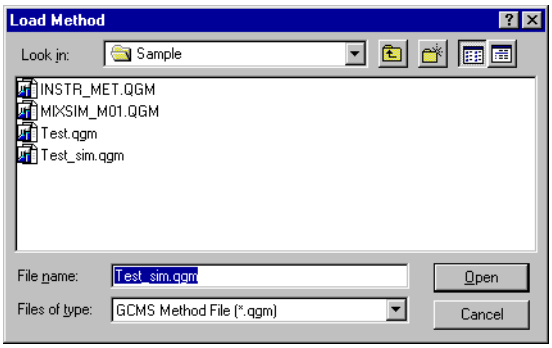


Figure 5.18 "Load Method" Dialog Box

4. Select the method file in which the calibration curve is saved, and click the **Open** button. The calibration curve and compound table are displayed.

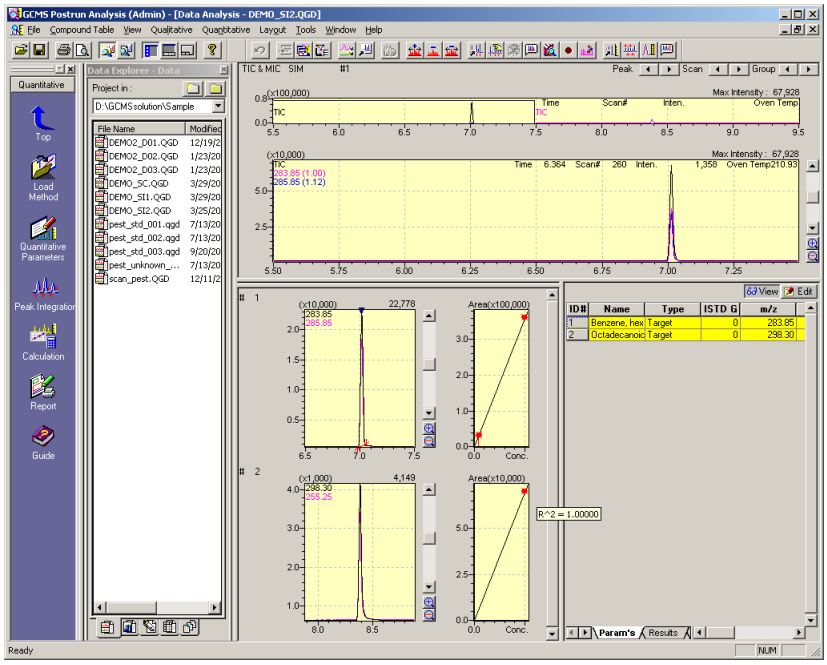


Figure 5.19 The Calibration Curve and Compound Table are Displayed

5. Click the **Quantitate for All IDs** toolbar button or select **Quantitative > Calculate**. After Quantitation, the results can be viewed.

6. Click the Results tab of the Compound Table to display the results of quantitation.

The information included in the result table is described in the following table.

Results Table

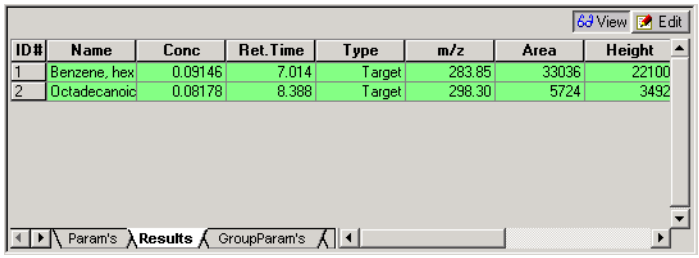


Figure 5.20 Results Table

Column	Description
ID#	Displays the compound ID number.
Name	Displays the name of the compound.
Conc	Displays the calculated concentration.
Ret. Time	Displays the retention time of the identified peak.
Type	Displays the type as entered in the compound table. If a peak is not identified, "Unknown" is displayed.
m/z	Displays the m/z value entered in the compound table.
Area	Displays the area of the identified peak.
Height	Displays the height of the identified peak.
Unit	Displays the unit selected in the Compound table.
Recovery	Displays the calculated percent recovery. The percent recovery is calculated by performing quantitation on a spiked sample. Yield calculation formula: (percent yield) = (calculated concentration of spiked sample - calculated concentration of unspiked sample)/(amount spiked) x 100 When creating the batch table, the unspiked and spiked samples must always be entered sequentially, with the unspiked sample preceding the spike sample. Enter the information for the unspiked sample, setting the batch table Sample Type to Unspiked. Then enter the information for the spiked sample and set its Sample Type to Spiked.
Mode	If peak integration has been performed displays the peak integration mode. • Auto: Indicates that automatic peak integration and identification have been performed. • Manual Integrate: Indicates that manual peak integration or identification has been performed.
Search	States if a Similarity Search was performed on the identified spectrum. To display the search results, double-click the cell or select Show Search Results for Compound Table from the right-click menu.
SI	The result of similarity calculations are displayed, when the pattern matching of identification parameters is checked and the standard spectrum is registered in the Compound table. In the FASST measurement interval, the similarity calculations are performed for the Scan spectrum.

Note

Compounds that have not been identified are displayed in gray rows. Identified compounds are displayed in green rows.

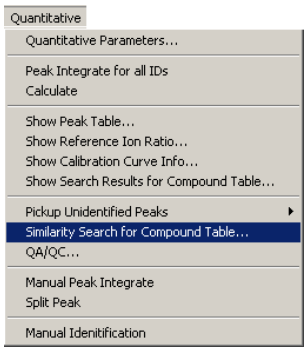
5.5 Similarity Search

This section describes how spectra in the library that are highly similar to the spectra for compounds in the compound table are listed and displayed. The spectra from the library, or candidate spectra, are displayed sequentially from the most similar to the compound in the compound table, or target compound. It also describes the comparison between candidate and target spectra.

5.5.1 Similarity Search for Compound Table

1. Open the "Data Analysis" window in the Quantitative or Compound Table mode. Click the Data tab in the Data Explorer, and double-click the appropriate data file icon. The compound table is displayed, as well as the chromatogram and calibration curves, if available.

2. Select the Similarity Search for Compound Table command from the Quantitative menu. The Compound Table Search tab of the "Quantitative Parameters" dialog box is displayed.



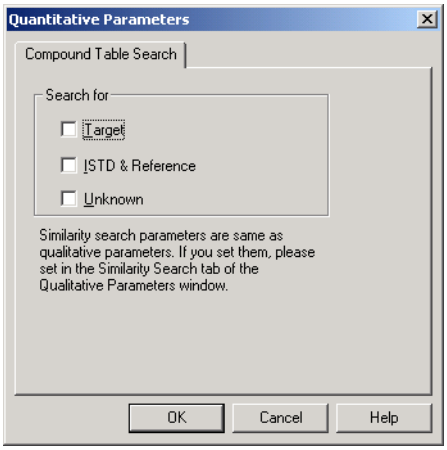


Figure 5.21 Quantitative Parameters Compound Table Search Tab

3. Select which types of compounds to include in the Similarity Search from Target, ISTD & Reference, and Unknown. More than one type may be included in the search at a time.

   After making your selection, click the **OK** button, and the Similarity Search is performed.

Note

If the target compound for a Similarity Search is acquired using the FASST, the Scan spectrum is used for the search.

4. To view the search results, double-click the row of the desired compound or select **Show Search Results for Compound Table** from the right-click menu. The "Similarity Search Results" window is displayed.

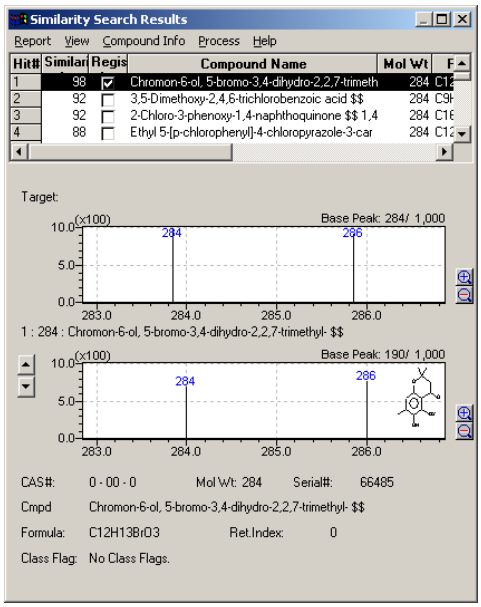
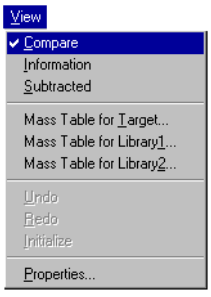


Figure 5.22 "Similarity Search Results" Window

Compounds are listed in the "Similarity Search Results" window sequentially, starting with compounds having the highest degree of similarity. The list can be scrolled with the buttons to the left of the middle spectrum. The middle spectrum changes to the spectrum of the compound that is currently selected in the list.

The top spectrum is the target spectrum, and the second spectrum is a candidate compound spectrum from the library. The information in the lower portion of the window varies depending on which display option is currently selected in the View menu. If Compare is selected, the lower portion contains a candidate spectrum from the same library as the second spectrum. If Information is selected, the compound information about the candidate spectrum is displayed. If Subtract is selected, the lower portion displays the resultant spectrum from subtracting the candidate spectrum from the target spectrum.



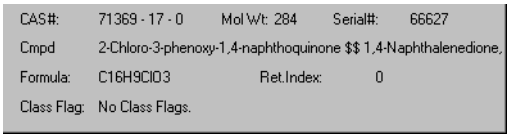


Figure 5.23 Information Selected in View Menu

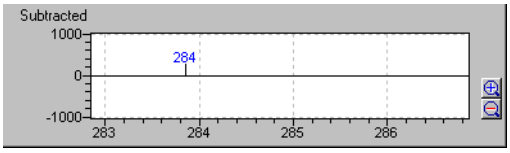


Figure 5.24 Subtract Selected in View Menu

Note

The library file used for the Similarity Search is selected in the Similarity Search tab of the Qualitative Parameters. To open the "Qualitative Parameters" dialog box, either click the **Qualitative Parameters** toolbar button or select **Qualitative > Qualitative Parameters**.

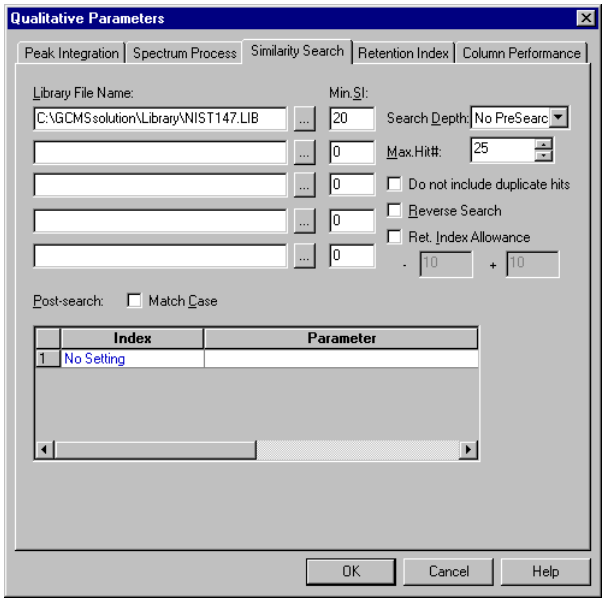


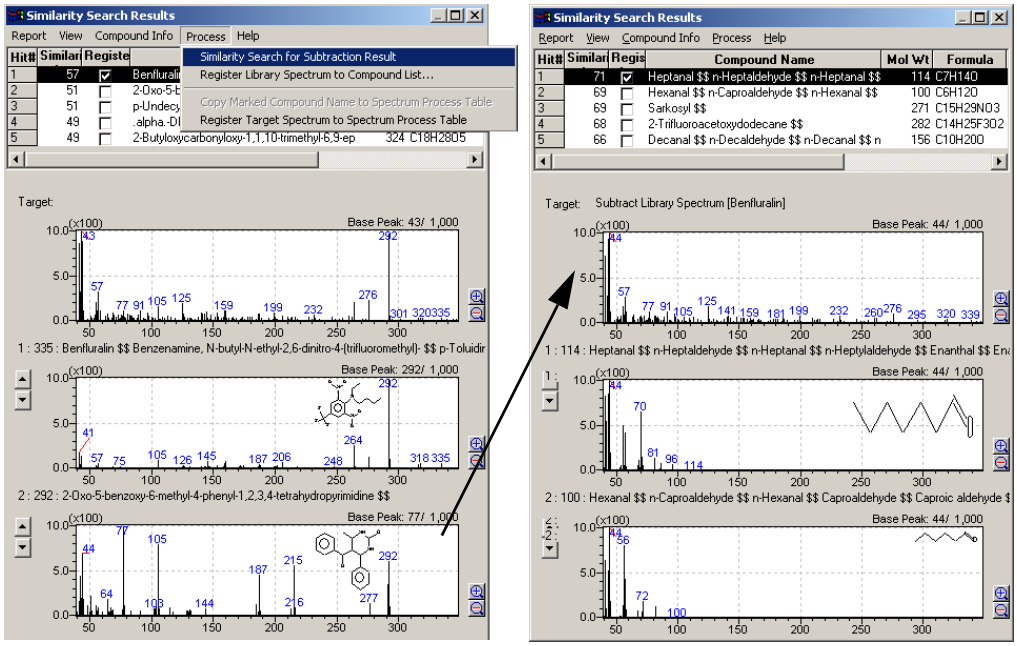
Figure 5.25 Library File Selection

Five library files may be selected under Library File Name. A minimum similarity level may be entered for each file under Min.SI. The maximum number of hits and other parameters are also specified in this tab. Refer to Appendix A "Peak Processing and Mass Spectrum Operations" on page 273 and GCMS Help for more information about the Similarity Search parameters.

5.5.2 To Execute a Similarity Search for Subtraction Result

When you know previously that the target spectrum consists of spectrum of multiple compounds, by subtracting the spectrum hit by similarity search and executing similarity search again, you can obtain more accurate search result.

1. Select **Process > Similarity Search for Subtraction Result** command.
2. A new Similarity Search window opens, and the subtracted spectrum and the search results are displayed.

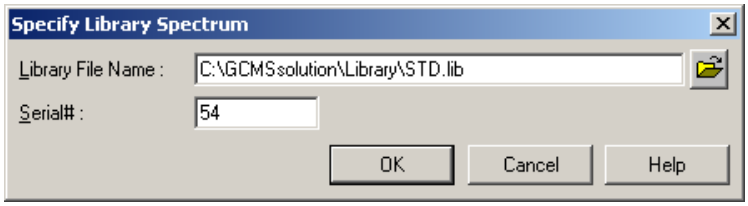


3. You can also execute a similarity search for the subtraction result again. (up to 2 times consecutively)

5.5.3 Specify Library Spectrum

You can add the library spectrum to the end of the hit list on Similarity Search window. By doing this, you can display the similarity search results of the spectrum and the similarity index, then also can execute Similarity Search for Subtraction Result.

1. Select the **Process > Register Library Spectrum to Compound List** command.
2. "Specify Library Spectrum" window is opened.
   Enter the library file name and serial number of spectrum which you want to add to the Hit List.



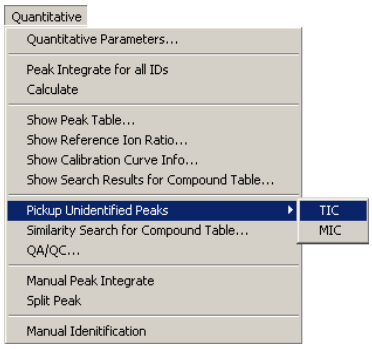
5.6 Analyzing Unknown Peaks

This section describes the method used to analyze the spectra of compounds listed in the compound table that are not target compounds. It is used primarily to analyze contaminants and other unknown components.

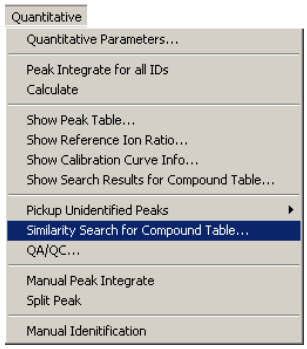
1. Open the "Data Analysis" window in the Quantitative or Compound Table mode. Click the Data tab in the Data Explorer, and double-click the appropriate data file icon.

   The compound table is displayed, as well as the chromatogram and calibration curves, if available.

2. Select TIC or MIC from Pickup Unidentified Peaks in the Quantitative menu. The unidentified peak is integrated and listed in the results table.



3. Select the Similarity Search for Compound Table command from the Quantitative menu. The Compound Table Search tab of the Quantitative Parameters is displayed.



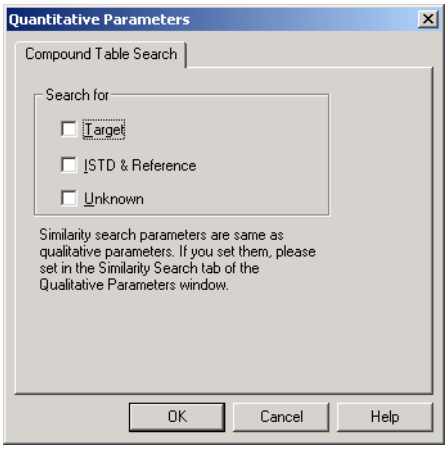


Figure 5.26 Quantitative Parameters Compound Table Search Tab

4. Select Unknown as the type of compound for which to search, and click the **OK** button. The Similarity Search is performed.

5. To view the search results, double-click the row of the desired compound or select **Show Search Results for Compound Table** from the right-click menu. The "Similarity Search Results" window is displayed.

5.7 Grouping

This section describes the method used to group compounds with similar characteristics and calculate the concentration for each group.

1. Click the **Calibration Curve** icon on the Assistant bar. The screen is displayed in Calibration Curve mode.

2. Load the method file. Click the Method tab in the Data Explorer, and double-click the icon of the method file to be loaded.

The Compound table registered in the loaded method file is displayed.

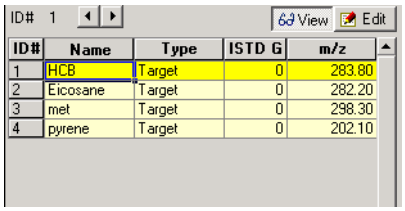


Figure 5.27 Compound Table

3. Double-click the Data tab in the Data Explorer and drag the data file for the calibration curve from the Data Explorer to Data File Tree.

4. Click the **Edit** button in the Compound table and change to the Edit mode. Create a group in the Compound table.

5. Click the Group Param's tab.

Enter the Group Name, Group Type, Conc, etc. For a calibration curve, the type of the grouping should be selected from the following:

Default: The grouping method selected in Quantitative parameters

Group calibration: Combines peak area and peak height of the compounds that are grouped, and quantitates the respective group using the calibration curve.

Concentration Sum: Draws the calibration curve for each compound, quantitates each compound, and combines concentrations of the compounds.

6. Click the Param's tab. Set the group number for respective compound selected in the Compound table.

7. Click the **View** button and switch to the View mode.

8. Click the **Peak Integration for All Data** icon on the assistant bar. The calibration curve is displayed.

9. The result of group quantitation is displayed on the Group Result tab in the Compound table.

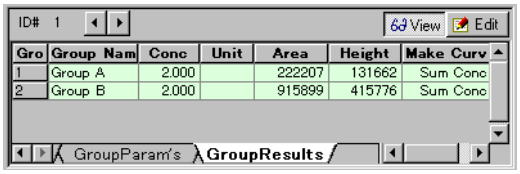


Figure 5.28 Compound Table

5.8 Printing Results

This section explains how to print the default quantitative analysis reports.

5.8.1 Calibration Curves

Display the "Calibration Curve" window, and open the appropriate method file. Verify that the correct data files are listed in the Data File Tree. To print a report of the calibration curve, select **File > Print Image > Print** or click the **Print** toolbar button. Select **File > Print Image > Preview**, or click the **Print Preview** toolbar button to view the report on-screen before printing. A default report format is used to print the calibration curves. To edit the default Image report format, select F**ile > Print Image > Edit Format.**

Print Image Report

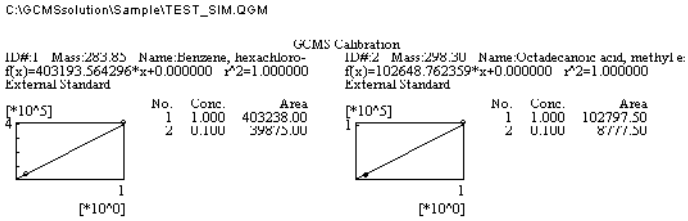


Figure 5.29 Print Image Preview

5.8.2 Custom Report

To print a custom report from an open data file in the "Data Analysis" window, select **File > Report**. The "Data Report" window is displayed. Either create a new report format and select the currently opened data file as the data file for each item, or open an existing report format file and select the currently opened data file as the data file for each item. Print the report. For information about creating custom report formats, refer to Chapter 6 "Generating Custom Reports" on page 165.

The procedure for generating a report from a report format template is described below.

1. Open the data file that will be used for the report in the "Data Analysis" window. Select **File > Report** to display the "Report" window.

2. Select **File > New Format File** to open the "File New" dialog box. Verify that the **Use Template** radio button is selected. The File New dialog box does not open unless "Prompt on File New" has been selected on the File New tab of the "Setting Options" window which is displayed by selecting Option in the Tools menu.

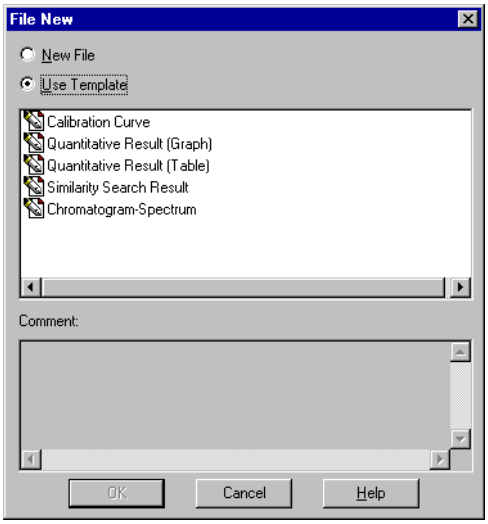


Figure 5.30 File New Dialog Box

3. Select a report format template from the list, and click the **OK** button. The formatted report, including the data from the open data file, is displayed in the "Data Report" window.

4. To print the report, use the **File > Print** command or click the **Print** toolbar button. A report generated with the Quantitative Result (Graph) template is illustrated below.

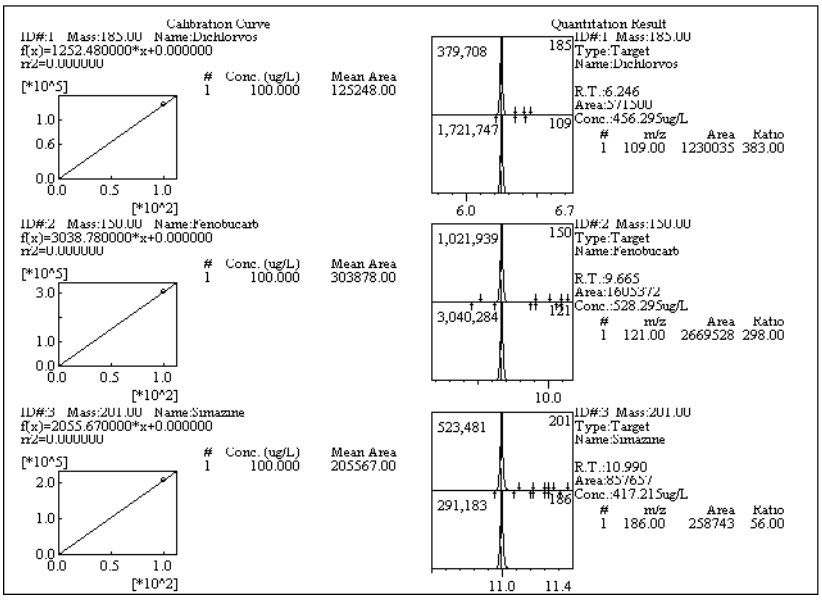


Figure 5.31 Report Generated with the Quantitative Result (Graph) Template

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6 Generating Custom Reports

6.1 Creating the Report Format

This section describes the procedure for creating a custom report format. This format may later be used to print data.

6.1.1 "Report" Window

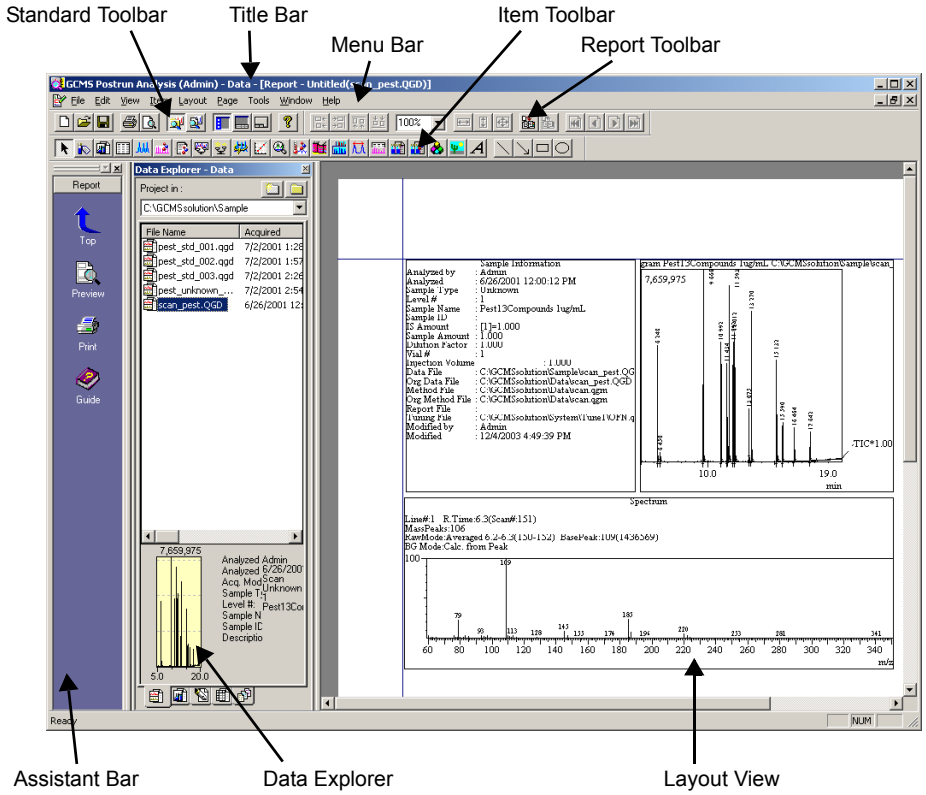


Figure 6.1 "Report" Window

Title Bar	Displays the name of the application, user, active window, and file.
Menu Bar	Displays the menus that are available in the active window.
Toolbar	Displays the command buttons that are available in the active window.
Assistant Bar	Displays the command icons that apply to a specific function or window. To open the window or activate the command, click the appropriate icon.
Data Explorer	Displays all files by type, including Data, Method, Report Format, Batch, and All Files. Double-clicking the file icon can open report Format files. A data file can easily be assigned to an item in the report by dragging it to the appropriate item.
Layout View	Displays the current item placement for a page of the report. Which items to include on the page, their location, and other properties may be set on this page.

6.1.2 Report Toolbar

1. Object Tools

Button	Name	Function
	Pointer	Moves and resizes items on the page.
	Sample Information	Inserts the information entered about the sample, such as the user name, sample name, sample type, and vial number, as well as other information, as specified in the Sample Information Properties.
	Method Content	Inserts the instrument parameters and data processing parameters, as specified in the Method Properties.
	Peak Table	Inserts the peak table as specified in the Peak Report Properties.
	Chromatogram	Inserts the chromatogram as specified in the Chromatogram Properties.
	Spectrum Graph	Inserts the spectrum and any additional information specified in the Spectrum Properties.
	Mass Table	Inserts the mass table specified in the Mass Table Properties.
	Quantitation Graph	Inserts the chromatograms and reference ion table, as well as other information specified in the Quantitation Properties.
	Quantitation Table	Inserts the quantitation results table as specified in the Quantitation Results Table Properties.
	Group Result	Inserts the group result of the data file to appear on the report.
	Calibration Curve	Inserts the calibration curve and table, as well as other information specified in the Calibration Properties.
	Tuning	Inserts the tuning conditions and results, as well as other information specified in the Tuning Properties.
	Status Log	Inserts a log of the MS status as specified in the Status Log Report Properties.
	Library Search	Inserts information about the target and candidate compounds as specified in the Library Properties.
	Spectrum Comparison	Inserts the comparison results as specified in the Spectrum Comparison Properties.
	Column Performance	Inserts the table specified in the Column Performance Report Properties.
	Spectrum Check	Inserts the spectrum check result of the data file to appear on the report.
	Summary (Conc.)	Displays the concentration lists of multiple data.
	Summary (Compound)	Displays the quantitation results of multiple data (Conc., Area, Height and etc.) lists for each compound.

Button	Name	Function
	Configuration	Inserts information about the system configuration as specified in the Configuration Control Properties.
	Picture	Inserts any graphic file, such as a company logo, as specified in the Picture Properties.
	Text	Inserts the text specified in the Text Properties.
	Line	Draws a line that may be further specified by its Shape Properties.
	Arrow	Draws an arrow.
	Rectangle	Draws a rectangle that may be further specified by its Shape Properties.
	Ellipse	Draws an ellipse that may be further specified by its Shape Properties.

2. Formatting Tools

Button	Name	Function
	Align Left	Aligns selected objects along their left edges at the furthest left edge.
	Align Right	Aligns selected objects along their right edges at the furthest right edge.
	Align Top	Aligns selected objects along their top edges at the highest edge.
	Align Bottom	Aligns selected objects along their bottom edges at the lowest edge.
	Zoom	Zooms in and out of the Layout View to the displayed percentage. Enter a percentage directly, or select one from the Zoom combo box.
	Make Same Width	Increases the width of the selected objects to match that of the widest item.
	Make Same Height	Increases the height of the selected objects to match that of the tallest item.
	Make Same Size	Increases the size of the selected objects to match the width of the widest item and the height of the tallest item.
	Insert	Adds a page to the report format. The new page is inserted after the currently displayed page.
	Delete	Removes the page that is currently displayed.
	First	If there are multiple pages, goes to the first page of the report format.

Button	Name	Function
	Previous	If there are multiple pages, goes to the previous page.
	Next	If there are multiple pages, goes to the next page.
	Last	If there are multiple pages, goes to the last page.

3. Standard Tools

Button	Name	Function
	Print	Prints the report.
	Preview	Opens the print preview screen so that the report can be verified before printing.
	Open	Opens existing report format files.
	Save	Saves the report format file. This overwrites the existing file; to save the file to a new name, use **File > Save Format File As**.

Note

To turn the toolbar into a floating palette, double-click the toolbar (the area around the command buttons), or click the toolbar and drag it to the desired location.

Figure 6.2 Floating Palette

This function helps you make the most efficient use of the screen. Use this function when the width of the tool bar must be reduced. To return the palette to the toolbar, either double-click the palette title bar or drag the palette into the toolbar.

6.1.3 Creating the Report Format File

1. Click Assistant Bar **Report Format** icon. The "Report" window opens and displays a new, untitled file.

2. After opening a new file, select **Page Setup** from the File menu. The "Page Setup" dialog box is displayed.

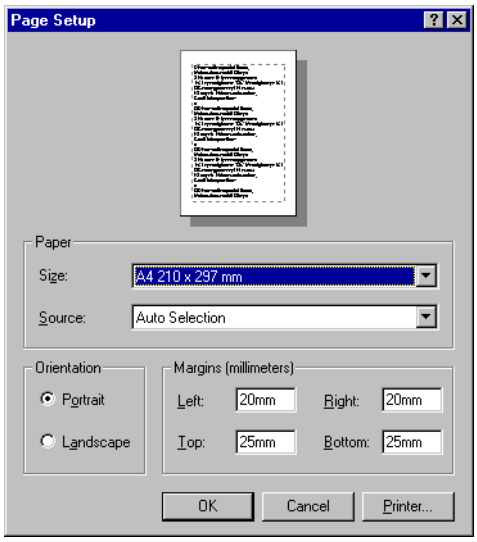


Figure 6.3 "Page Setup" Dialog Box

3. Enter the following parameters.

Group	Parameter	Description
Paper	Size	Select which size paper to use for printing.
	Source	Select the paper tray or how the paper is supplied to the printer.
Orientation	Portrait or Landscape	Determine whether to use the paper horizontally (landscape) or vertically (portrait).
Margins (millimeters)	Left, Right, Top, and Bottom	Enter how much space to leave from the left, right, top, and bottom of the page in millimeters.
	Printer	Select which printer to use by default.

4. Click a toolbar item button, or select an item from the Item menu. Drag the cursor over the desired location in the Layout View to place the item in the report.

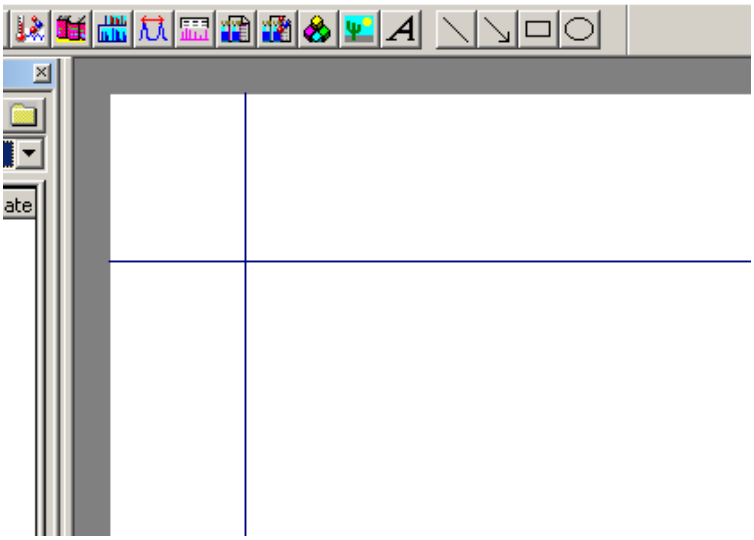


Figure 6.4 Layout View

The appropriate properties dialog box is displayed.

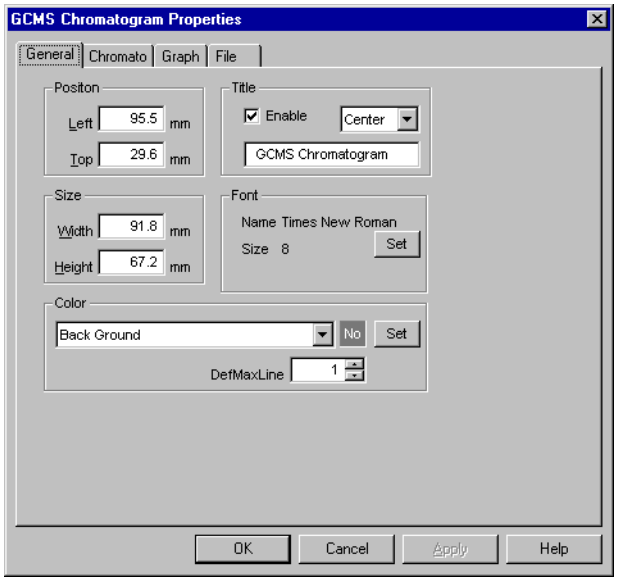


Figure 6.5 Properties Dialog Box

5. Set the parameters in the various **Properties tabs**. Although most of the parameters vary by item, each item has a **General tab** for setting the position, size, title, font and color, and a **File tab** for determining the source data file. After all of the parameters are entered, click the **OK** button. The properties dialog box is closed, and the entered parameters are applied.

   Add additional items to the report, and enter their parameters, as necessary.

6. If the report will be composed of multiple pages, click the **Insert** button to add a page after the page that is currently displayed. 

7. Select **View > Header/Footer**. The "Header/Footer" dialog box is displayed.

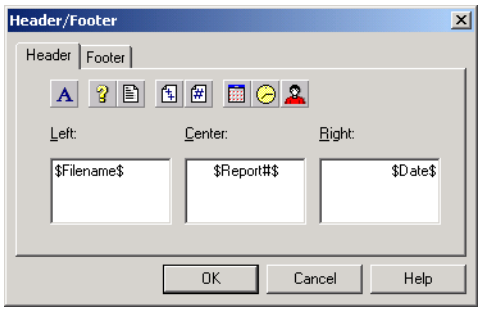


Figure 6.6 Header/Footer Dialog Box

8. The contents of the table below may be aligned in the Left, Center or Right of the page, as determined by the text box in which they are entered. Enter information to be printed at the top of the report in the **Header tab** and the information to be printed at the bottom of the report in the **Footer tab**.

   Place the cursor in the Left, Center, or Right text box, and click the button to enter the variable for the associated text.

Button	Name	Function	Variable
	Font	Click the **Font** button to open the "Font" window. Select the font, including size, style and color, in which to display the header or footer, and click OK.	
	Version	Click the **Version** button to include the version of GCMSsolution Ver. 2 in the header or footer text.	$Version$
	Filename	Click the **Filename** button to include the name of the data file in the header or footer text. When using different data for each item, include the data file name with the item.	$Filename$
	Report#	Click the **Report#** button to include the number of the report in the header or footer text. Reports are numbered consecutively as they are printed. Enter the number for the first report in the "Report Option" dialog box.	$Report#$
	Page#	Click the **Page#** button to include the page number in the header or footer text.	$Page#$
	Date	Click the **Date** button to include the date, as formatted by Windows, in the header or footer text.	$Date$
	Time	Click the **Time** button to include the time, as formatted by Windows, in the header or footer text.	$Time$
	Username	Displays user name.	$User$

9. After setting the properties for all included items, and entering header and footer text, click the **Preview** button to view the printed report.

Button	Command
Print	Print the report. Refer to Section 6.2.3 "Printing the Report", page 173.
Next Page	Preview the next page.
Prev Page	Preview the previous page.
Two Page	Preview two pages simultaneously.
One Page	Preview one page at a time.
Zoom In	Enlarge the previewed report.
Zoom Out	Reduce the previewed report.
Close	Exit the preview window.

10. Save the newly created format as a report format file.

Click the **Save** toolbar button.

The "Save As" dialog box is displayed the first time that the report format is saved.

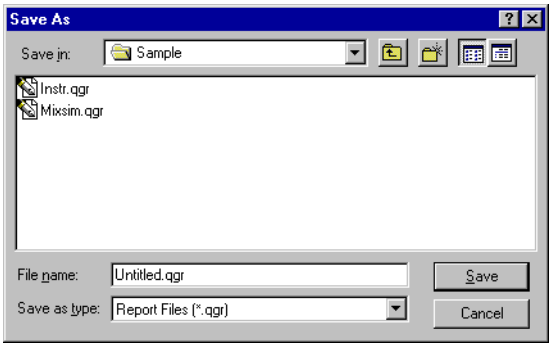


Figure 6.7 "Save As" Dialog Box

Select the directory where the file will be saved, and enter a file name. Click the **Save** button, and the report format is saved as a report format file (*.qgr).

Use the **Save** button to save modifications to a report format file by overwriting the existing file. Refer to Section 6.2.1 "Opening the Report Format File", page 173.

Note

Use **File > Save Format File As** to save the file under a new name unless the existing file is to be overwritten with the modified file. The "Save As" dialog box is displayed. Select a directory where the file will be saved, enter a file name, and click the **Save** button.

6.2 Using Report Format Files

This section describes the procedure for opening existing report format files, importing data, and printing reports.

6.2.1 Opening the Report Format File

1. Click the Assistant Bar **Report Format** icon. The "Report" window is displayed.

2. Open the appropriate report format file with **File > Open Format File**, **Open** toolbar button, or Data Explorer. The report format file is displayed in the Layout View.

6.2.2 Importing Data into the Report Format File

1. Click the Data tab of the Data Explorer. All of the data files for the currently selected project are listed.

2. Click the appropriate data file in the Data Explorer, and drag it to the Layout View. The data file is loaded into the report, and the data is displayed.

   Data may also be imported by selecting **File > Load Data File**.

6.2.3 Printing the Report

1. Click the Assistant Bar **Print** icon to display the "Print" dialog box.

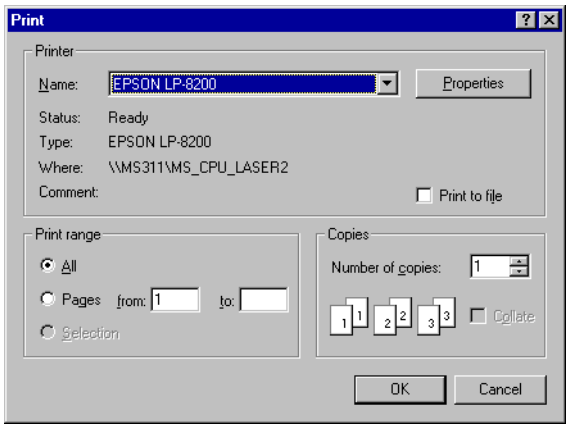


Figure 6.8 "Print" Dialog Box

Note

Clicking the **Properties** button next to the name of the Printer changes the printer parameters. For details about the printer properties, refer to the printer manual.

2. Select which pages to print from "All", "Pages" or "Selected". "All" prints every report page. "Pages" allows a range of pages to be printed. "Selected" prints portions of the report that were selected before the print command was executed.

3. Enter the number of copies to print in Number of copies.

4. Click the **OK** button to print the report.

7 Continuous Analysis

7.1 Overview

This section describes two types of automated processing. One is used by GCMS Real Time Analysis to acquire and analyze data from multiple samples. The other is used by GCMS Postrun Analysis to analyze the data from multiple data files. For both types of automation, a method file must first be created.

7.1.1 Automated Data Acquisition and Data Analysis

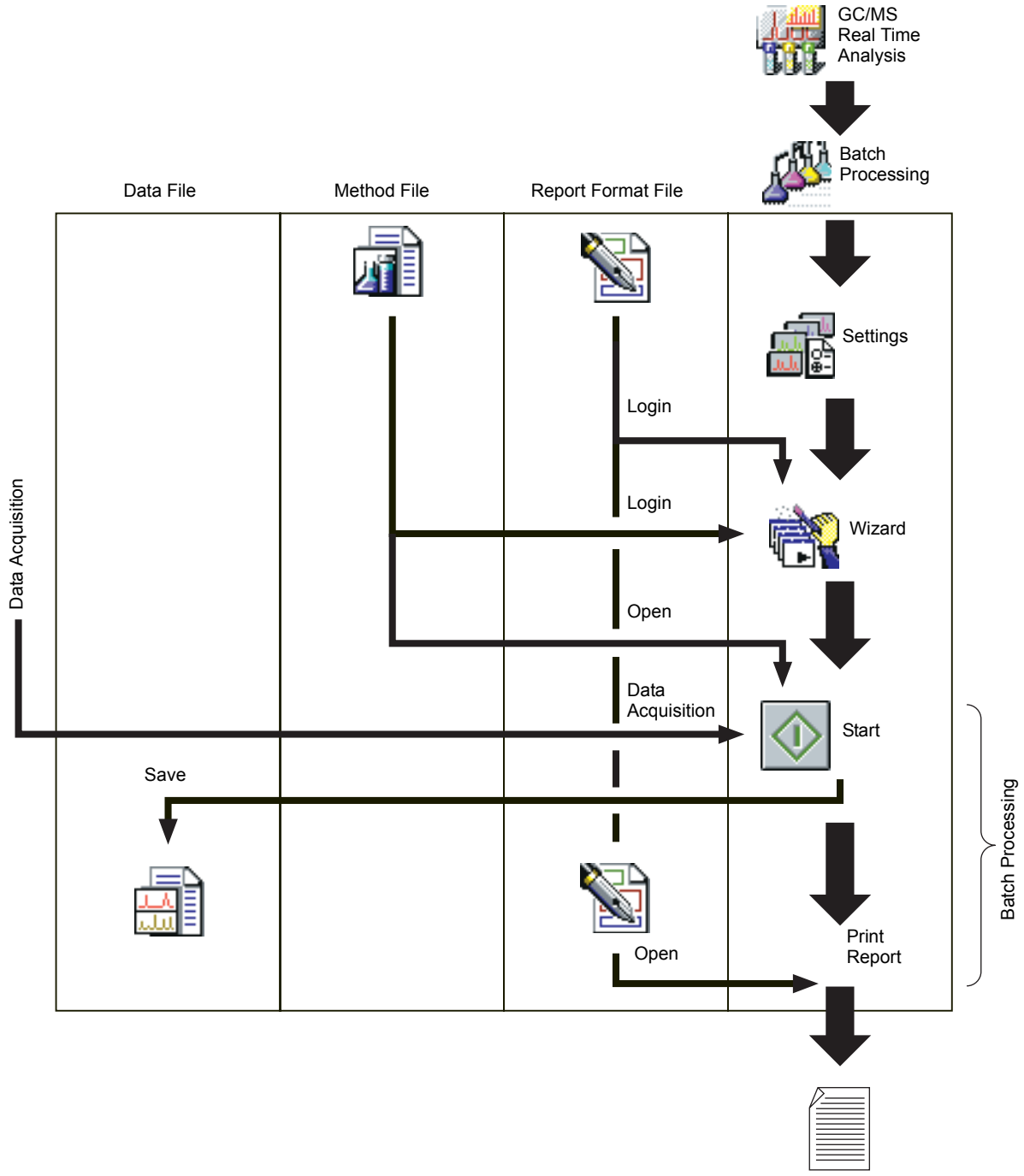


Figure 7.1 Automated Data Acquisition and Analysis

7.1.2 Automated Postrun Analysis

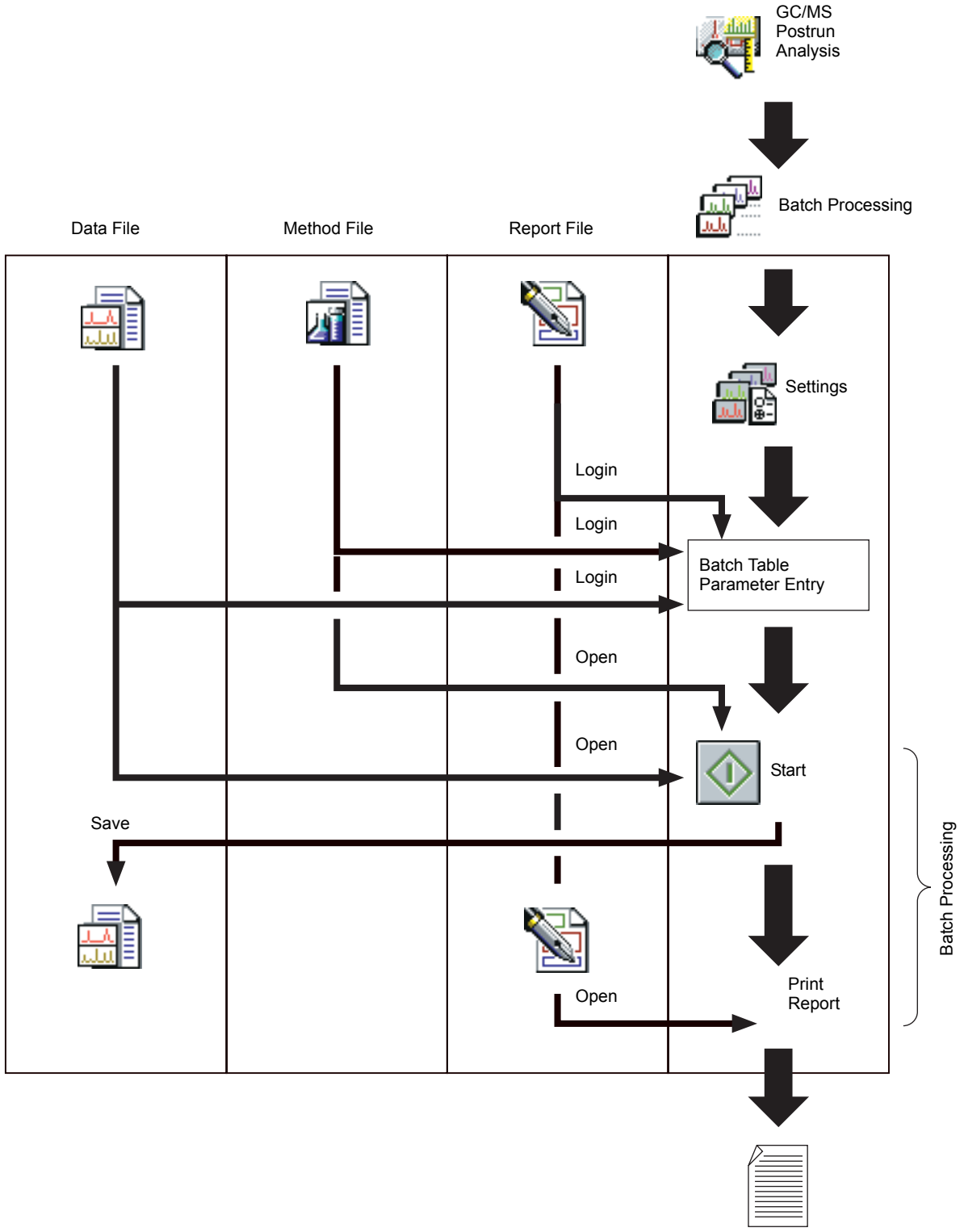


Figure 7.2 Automated Postrun Analysis

7.2 Automated Data Acquisition and Data Analysis

This section describes data acquisition and analysis with GCMS Real Time Analysis using an autosampler. For information about the qualitative and quantitative analysis parameters, refer to
Chapter 4 "Qualitative Analysis" on page 111 and Chapter 5 "Quantitative Analysis" on page 137.

7.2.1 Creating a Batch Table

1. Start GCMS Real Time Analysis, and click the Assistant Bar **Batch Processing** icon. The "Batch Table" window is displayed.

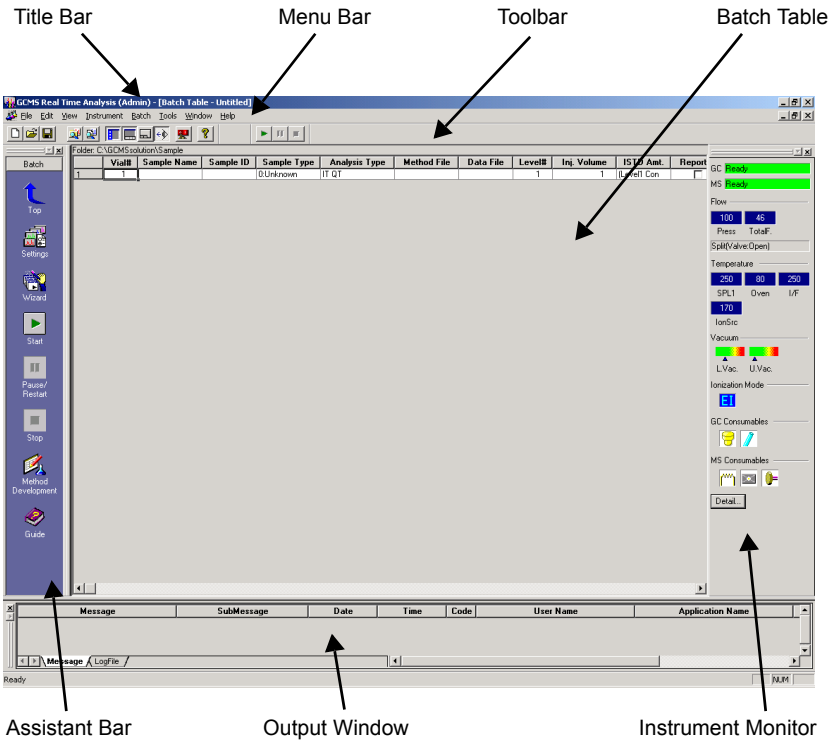
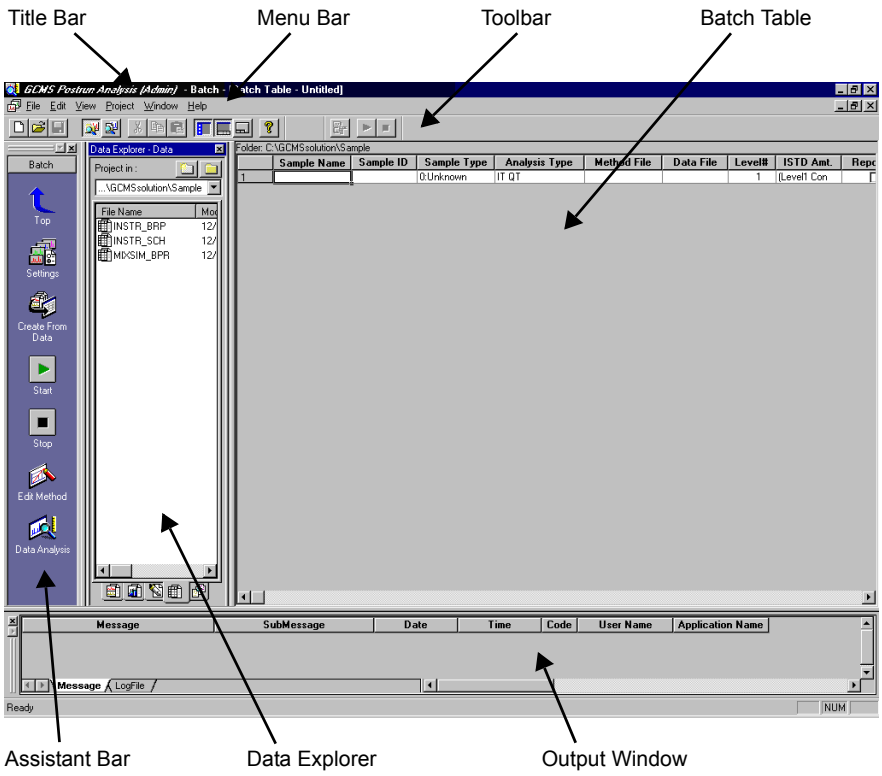


Figure 7.3 "Batch Table" Window



2. Click the Assistant Bar **Settings** icon, and the "Settings" dialog box is displayed.

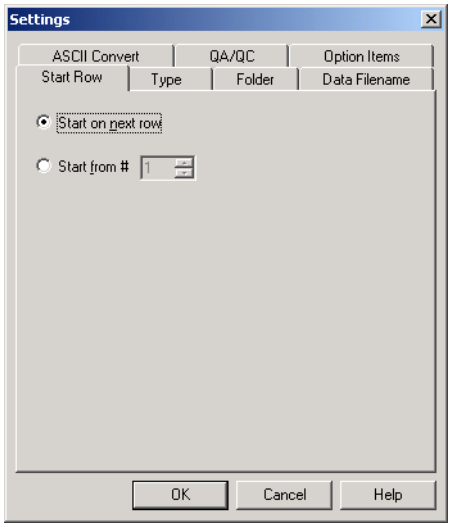


Figure 7.4 "Settings" Dialog Box

Start Row Tab

Indicate the row where batch processing will start.

Parameter	Description
Start on next row	Batch processing starts from the row after processing was stopped. If a data file was not created for that row, batch processing will start on that row. Before processing starts, a message is displayed to verify whether to start from the next row or the first row.
Start from #	Batch processing starts from the indicated row. Before processing starts, a message is displayed to verify whether to start from the indicated row or the first row.

Type Tab

Select the line for batch data acquisition and post run analysis.

Parameter	Description
Line 1 Line 2 Line 1 & Line 2	Selects which line to use in the Batch table. Line 2 and Line 1 & 2 are only selectable when multiple analytical lines are enabled in the system configuration. When Line 1 & 2 is selected, two rows at a time are added to (or deleted from) the batch table. In this way the processing of both Lines 1 and 2 is performed for each sample indicated.

Folder Tab

This tab determines the directory to search by default for a type of file. For example, if the name of a data file, but not a full path, is entered, the program will get the full path from the folder specified in this tab.

Parameter	Description
Use Current Folder	The program uses the folder that is currently selected in the Data Explorer. This is usually the folder in which the batch file was saved.
Use Specified Folder	Allows the entry of the default directory for the data, method, and report format files. To browse for a directory, click the **Folder** button to the right of the Data File, Method File, and Report Format File text boxes.
Use Same Folder	To use the same directory for data, method and report format files, check Use Same Folder. The folder indicated in the Data File text box is used as the default folder for all of the files.

Data Filename Tab

Parameter	Description
Generates filename automatically with	When checked, the file name is automatically named using the information selected in the Selected Items box. Select the item(s) to be used in the name from the left box, and click the **Add** button to move the item of the Selected Items box. To remove an item from the name, select it in Selected Items, and click the **Del** button. Determine the order in which the items appear in the name by selecting the item in the Selected Items box and clicking the **Up** and **Down** buttons. Items that may be included in the name: Batch Filename, Batch Table Line #, Method Filename, Username, Sample Name, Sample ID, and Current Date.
Auto-increment format	Selects one of the four auto-increment formats. The options include: 1, 2, ...; 01, 02, ...; 001, 002, ...; and 0001, 0002, ...

ASCII Convert Tab

When Output ASCII File(s) is checked, the results of batch processing are exported to an ASCII file.

Parameter	Description
Output per batch Output per analysis	Indicates whether to export the selected information to a text file after processing the entire batch (Output per batch) or one row of the batch table (Output per analysis).
Output File	Allows the entry of the full path name into the Output File text box, or filename selection by browsing for the appropriate directory and entering the name by clicking the **Folder** button to the right of the text box.
Over write Auto-increment	Indicates whether to overwrite an existing text file or to increment the filename if a file with the same name already exists.
Output Items	Selects the items to include in the text file from the following: Data File Properties, Compound Quantitative Result, Compound Search Result, Qualitative Peak Table, Column Performance Table, Spectrum Process Table, Spectrum Search Result, Chromatogram (TIC), Chromatogram (MIC), and Spectrum. Checked items are exported to the text file.
Delimiter	Used to determine how to separate the information in the text file. The options include Tab, Comma and Other. If other is selected, enter the delimiter in the text box to the right.

QA/QC Tab

Check Execute QA/QC to perform QA/QC.

Parameter	Description
Output File (Text Style)	Allows the entry of the full path name into the Output File text box, or filename selection by browsing for the appropriate directory and entering the name by clicking the **Folder** button to the right of the text box. The output files are tab delimited.
Merge File, Overwrite File, Auto-increment	Indicates whether to add to the file (Merge File), overwrite it (Overwrite File), or make new files by adding a number to the file name automatically, if a file with the same name already exists.
Output HTML Style File (<File name>.htm)	When checked, exports the QA/QC results to an HTML file in addition to text file output. The HTML file has the same name indicated in the Output File text box, with an ".htm" extension.
Output CSV Style File (<File name>.csv)	Selects whether to output CSV type files in addition. The CSV file has the same name indicated in the Output File text box, but with an ". csv" extension.

Option Items Tab

The batch table can include up to five Option columns as established by the Table Style. If these Option columns are included, use the Option Items tab to give the column a title. Up to 31 characters may be entered. The title may be referred to in the data file properties or in the sample information of a report.

3. Click the Assistant Bar **Wizard** icon, and Batch Table Wizard is displayed.

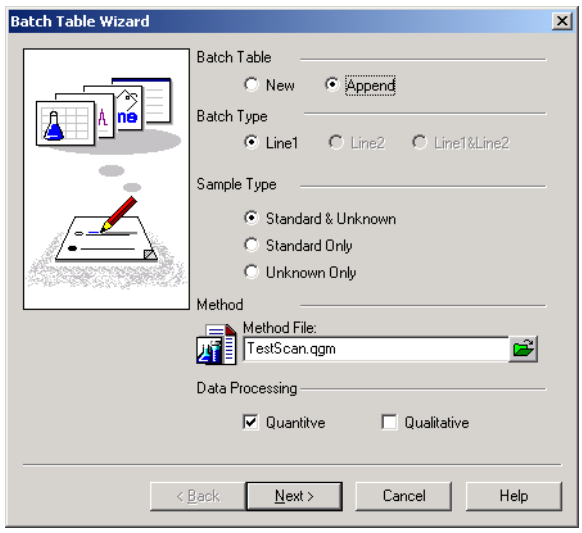


Figure 7.5 Batch Table Wizard - Initial Screen

Batch Table Wizard - Initial Screen

Parameter	Description
Batch Table	Selecting **New** creates a new table. **Append** adds to the last row of the currently displayed batch table.
Batch Type	Selects which Analytical Line to use in the Batch table. Some types cannot be entered depending on the system configuration. Select from Line 1 & 2, or Line 1 or Line 2 only.
Sample Type	Selects the type of samples that will be included in the batch. • Choose Standard & Unknown when both standards and unknown samples will be included in the batch. After clicking the **Next** button, enter the standard, then unknown information. The standard rows will be placed before the unknown samples in the batch table. If Quantitative is selected for Data Processing under Method, a new calibration curve will be generated based on the standard analysis results. The calibration curve is saved in the method file. The updated method will be used for subsequent analyses, and the unknown sample analysis results are quantitated using that calibration curve. • Select Unknown Only to create a batch table with unknown samples only. After clicking the **Next** button, the unknown sample parameter screen is displayed. Only unknown sample rows will be created in the batch table.

Parameter	Description
Method	Allows the entry of the full path name into the Output File text box, or filename selection by browsing for the appropriate directory and entering the name by clicking the **Folder** button to the right of the text box. If a Method file is currently open in the "Data Acquisition" window, its Method file name is displayed by default. The method file indicated here is entered in the batch table by default, but a different file can be specified in the table.
Data Processing	Specifies whether to perform quantitative and/or qualitative analysis. If Quantitative is checked, the Analysis Type will include Integration for Quantitative and Quantitative Calculations. If Qualitative is checked, the Analysis Type will include Integration for Qualitative, Make Spectrum Process Table, and Similarity Search.

After all of the parameters have been entered, click the **Next** button.
If Standard & Unknown or Standard Only is selected as the Sample Type, the Batch Table Wizard proceeds to the Standard Sample (1) screen. If Unknown Only is selected, the Batch Table Wizard proceeds to the Unknown Sample (1) screen.

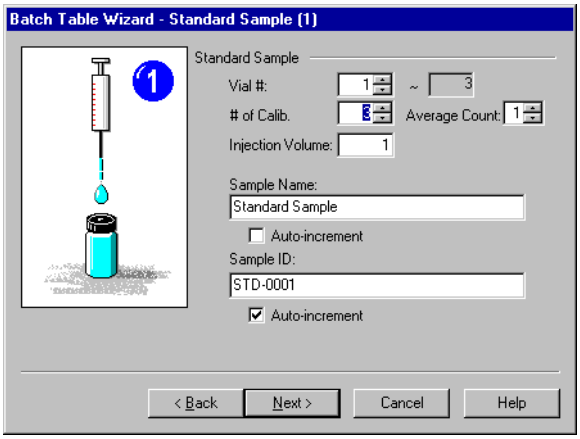


Figure 7.6 Batch Table Wizard - Standard Sample (1) Screen

Batch Table Wizard - Standard Sample (1)

This screen is displayed when "Standard & Unknown" or "Standard Only" is selected as the Sample Type in the first Batch Table Wizard screen.

Parameter	Description
Vial #	If an autosampler is used, enter the position number from the rack for the first standard vial. The text box to the right indicates the final vial position, calculated from the number of currently indicated calibration points. Ensure that the standard samples are correctly placed in the autosampler tray. This parameter is needed only when an autosampler is used.
# of Calib	The number of points in the calibration curve is determined from the method file and displayed as the default. This number can be changed in the Wizard. Changing the number of calibration points changes the final vial position indicated in Vial #.
Average Count	Specifies the number of injections per standard vial. The total number of standard analyses is the # of Calib. multiplied by the Average Count.
Injection Volume	Used to enter the injection volume. Once the Wizard is finished, the volume in each row of the batch table can be changed.
Sample Name	Specifies a Sample Name for the standards. This parameter is used in combination with Sample ID to distinguish a particular file of samples and standards from others. This parameter is optional.
Auto-increment	When Auto-increment is checked, the Sample Name is automatically incremented with a number, creating unique sample names for subsequent rows.
Sample ID	Specifies a Sample ID. This parameter is used to distinguish samples and is useful when analyzing the same sample using different Methods or when the same Method is used for the analysis of several samples. This parameter is optional.
Auto-increment	When Auto-increment is checked, the Sample ID is automatically incremented with a number, to create unique sample IDs for subsequent rows.

After all parameters have been entered, click the **Next** button.

The Batch Table Wizard proceeds to the Standard Sample (2) screen.

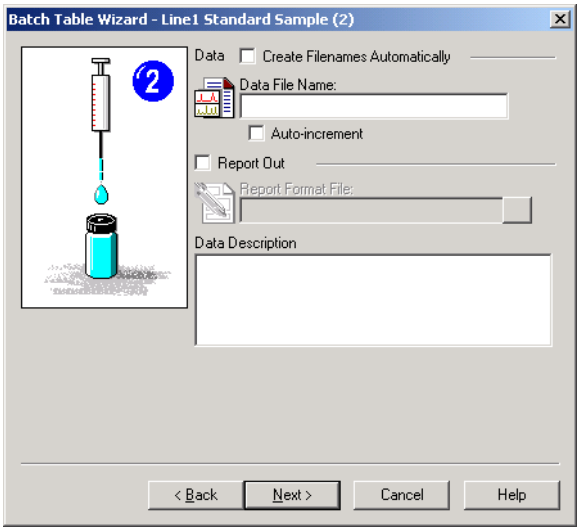


Figure 7.7 Batch Table Wizard - Standard Sample (2) Screen

Batch Table Wizard - Standard Sample (2)

This screen is displayed when "Standard & Unknown" or "Standard Only" is selected as the Sample Type in the first Batch Table Wizard screen.

Parameter	Description
Create Filenames Automatically	Specifies whether to automatically generate file names. This parameter can only be changed when a new batch table is created. When adding to an existing batch table, the current setting is indicated. Data file names cannot be defined when this parameter is checked.
Data File Name	Specifies the data file name. Select Auto-increment to automatically increment the file extension. Each data file name must be unique.
Report Out	Specifies whether to generate a report. If the item is selected, specify a Report Format file.
Report Format File	When printing a report, this item specifies the Report Format file. The file folder icon may be used to browse for the Report Format file.
Data Description	Allows a description to be entered in the data file, as desired.

After the parameters are entered, click the **Next** button.

The Batch Table Wizard proceeds to the Unknown Sample (1) screen.

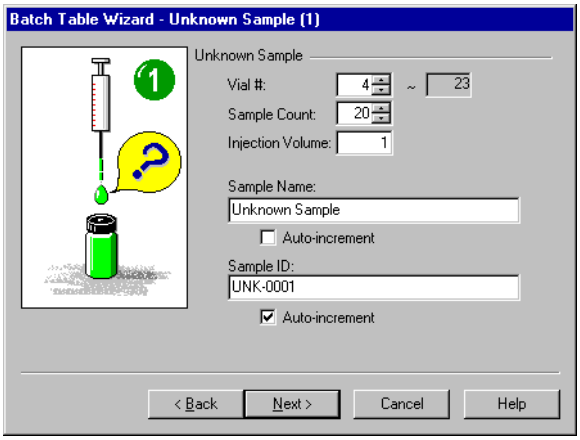


Figure 7.8 Batch Table Wizard - Unknown Sample (1) Screen

Batch Table Wizard - Unknown Sample (1)

This is the first Wizard screen to set up unknown samples in the batch table.

Parameter	Description
Vial #	If an autosampler is used, enter the position number of the first sample vial. The text box to the right indicates the final vial position as calculated from the currently displayed Sample Count number. Ensure that the samples are correctly placed in the autosampler tray. This parameter is needed only when an autosampler is used.
Sample Count	Used to enter the number of unknown samples for which data is to be acquired. If there are no unknown samples, enter 0, the **Next** button changes to the **Finish** button and there are no further parameters to enter.
Injection Volume	Used to enter the injection volume. Once the Wizard is finished, the volume in each row of the batch table can be changed.
Sample Name	Specifies a Sample Name for the unknown samples. This parameter is used in combination with Sample ID to distinguish a particular file of samples and standards from others. This parameter is optional.
Auto-increment	When Auto-increment is checked, the Sample Name is automatically incremented with a number, so that unique sample names are created for subsequent rows.
Sample ID	Specifies a Sample ID. This parameter is used to distinguish samples and is useful when analyzing the same sample using different Methods or when the same Method is used for the analysis of several samples. This parameter is optional.
Auto-increment	When Auto-increment is checked, the Sample ID is automatically incremented with a number, so that unique sample IDs are created for subsequent rows.

After entering the parameters, click the **Next** button.

The Batch Table Wizard proceeds to the Unknown Sample (2) screen.

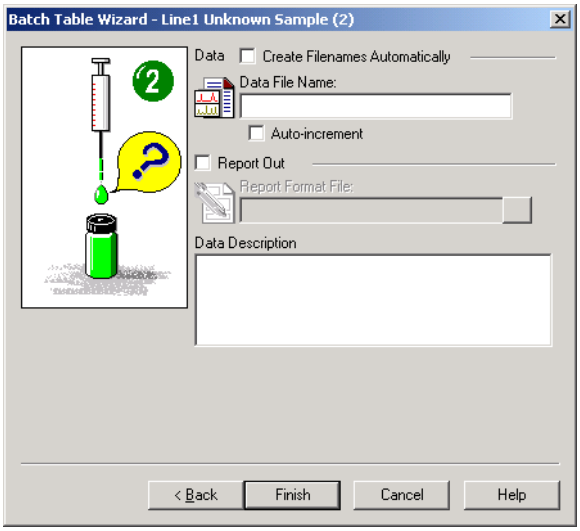


Figure 7.9 Batch Table Wizard - Unknown Sample (2) Screen

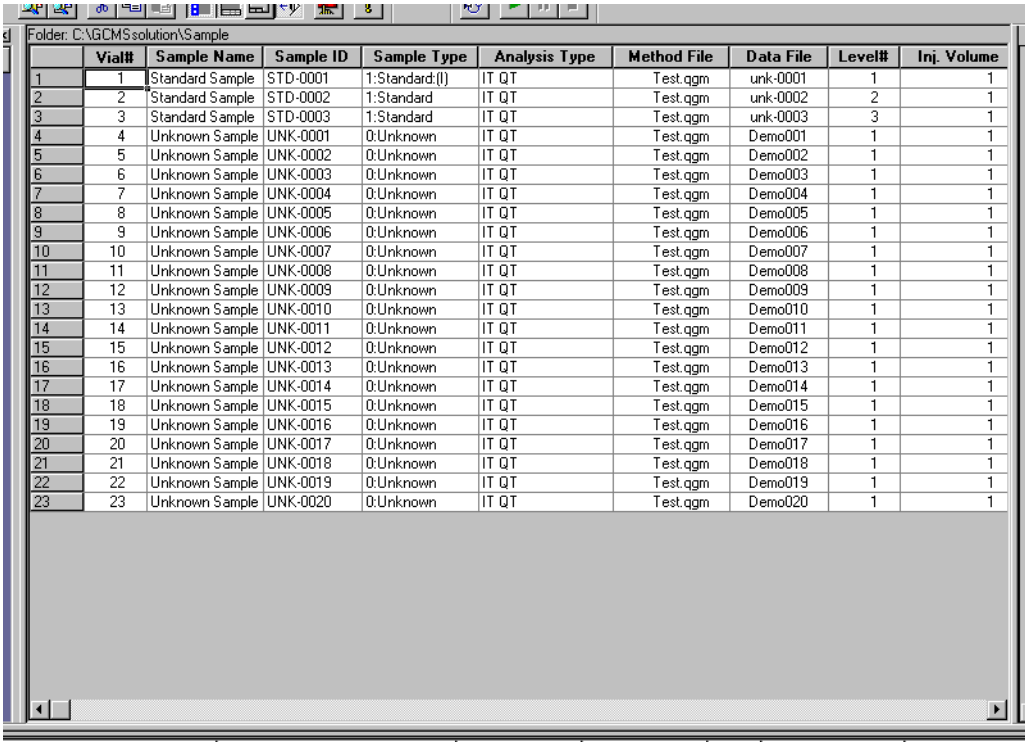
Batch Table Wizard - Unknown Sample (2)

This screen is displayed if Unknown samples are entered into the Batch Table Wizard Unknown Sample (1) screen.

Parameter	Description
Create Filenames Automatically	Specifies whether to automatically generate file names. This parameter can only be changed when a new batch table is created. When adding to an existing batch table, the current setting is indicated. Data file names cannot be defined when this parameter is checked.
Data File Name	Specifies the data file name. Select Auto-increment to automatically increment the file extension. Each data file name must be unique.
Report Out	Specifies whether to generate a report. If the item is selected, specify a Report Format file.
Report Format File	When printing a report, this item specifies the Report Format file. The file folder icon may be used to browse for the Report Format file.
Data Description	Allows a description to be entered in the data file, as desired.

After entering the parameters, click the **Finish** button. The Batch Table Wizard closes and a batch table is generated.

When parameters for specific rows differ from the Wizard parameter entries, make the changes directly in the batch table.



Folder: C:\GCMSsolution\Sample

	Vial#	Sample Name	Sample ID	Sample Type	Analysis Type	Method File	Data File	Level#	Inj. Volume
1	1	Standard Sample	STD-0001	1:Standard:(I)	IT QT	Test.qgm	unk-0001	1	1
2	2	Standard Sample	STD-0002	1:Standard	IT QT	Test.qgm	unk-0002	2	1
3	3	Standard Sample	STD-0003	1:Standard	IT QT	Test.qgm	unk-0003	3	1
4	4	Unknown Sample	UNK-0001	0:Unknown	IT QT	Test.qgm	Demo001	1	1
5	5	Unknown Sample	UNK-0002	0:Unknown	IT QT	Test.qgm	Demo002	1	1
6	6	Unknown Sample	UNK-0003	0:Unknown	IT QT	Test.qgm	Demo003	1	1
7	7	Unknown Sample	UNK-0004	0:Unknown	IT QT	Test.qgm	Demo004	1	1
8	8	Unknown Sample	UNK-0005	0:Unknown	IT QT	Test.qgm	Demo005	1	1
9	9	Unknown Sample	UNK-0006	0:Unknown	IT QT	Test.qgm	Demo006	1	1
10	10	Unknown Sample	UNK-0007	0:Unknown	IT QT	Test.qgm	Demo007	1	1
11	11	Unknown Sample	UNK-0008	0:Unknown	IT QT	Test.qgm	Demo008	1	1
12	12	Unknown Sample	UNK-0009	0:Unknown	IT QT	Test.qgm	Demo009	1	1
13	13	Unknown Sample	UNK-0010	0:Unknown	IT QT	Test.qgm	Demo010	1	1
14	14	Unknown Sample	UNK-0011	0:Unknown	IT QT	Test.qgm	Demo011	1	1
15	15	Unknown Sample	UNK-0012	0:Unknown	IT QT	Test.qgm	Demo012	1	1
16	16	Unknown Sample	UNK-0013	0:Unknown	IT QT	Test.qgm	Demo013	1	1
17	17	Unknown Sample	UNK-0014	0:Unknown	IT QT	Test.qgm	Demo014	1	1
18	18	Unknown Sample	UNK-0015	0:Unknown	IT QT	Test.qgm	Demo015	1	1
19	19	Unknown Sample	UNK-0016	0:Unknown	IT QT	Test.qgm	Demo016	1	1
20	20	Unknown Sample	UNK-0017	0:Unknown	IT QT	Test.qgm	Demo017	1	1
21	21	Unknown Sample	UNK-0018	0:Unknown	IT QT	Test.qgm	Demo018	1	1
22	22	Unknown Sample	UNK-0019	0:Unknown	IT QT	Test.qgm	Demo019	1	1
23	23	Unknown Sample	UNK-0020	0:Unknown	IT QT	Test.qgm	Demo020	1	1

Figure 7.10 Batch Table

4. Batch tables can be created and existing tables edited in the "Batch Table" window, without using the Batch Table Wizard.

If a batch table is created without the Wizard, the new table has only a single row initially. When the last row of the table is edited, a new, blank row is added automatically. A batch table can contain up to 1000 rows.

Pop-up Menus

Right-click the cell of interest to display the relevant pop-up menu. Select the appropriate command in the menu. The available commands range from copying and pasting to editing the Table Style.

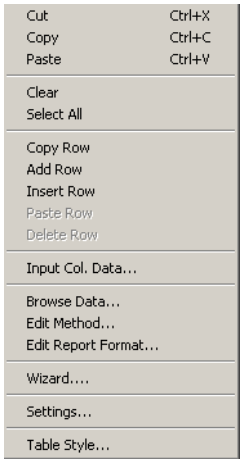


Figure 7.11 Right-click Menu

Sequential Entry

When information such as the vial number, sample name, sample ID, and data file are sequential for a number of rows, multiple rows can be entered by incrementing the information automatically. Highlight the appropriate column and select **Edit > Input Col. Data**. For example, to fill out the Sample ID column automatically, the following dialog box is displayed.

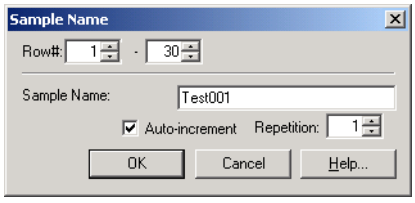


Figure 7.12 Automatically Filing the Sample ID Column

In the pictured "Sample Name" dialog box, clicking the **OK** button enters sample IDs from test 001 to test 030 in lines 1 - 30.

Changing the Batch Table Columns

To hide or show table columns, or change the order in which they appear, open the "Table Style" dialog box with the **Edit > Table Style** command or select **Table Style** from the right-mouse menu.

For more information about batch tables, refer to GCMS Help.

7.2.2 Performing Continuous Analysis

This section explains starting and stopping batch processing. Other batch table commands included on the Assistant Bar when in Batch Processing mode are also explained.

1. Start

Click the Assistant Bar **Start** icon, **Start** toolbar button or select **Batch > Start** to initiate batch processing. Batch processing starts with the first row or the row indicated in the Settings Start Row tab.

2. Pause

To pause batch processing, click the Assistant Bar **Pause/Restart** icon or **Pause/Restart** toolbar button, or select **Batch > Pause/Restart**. Continuous data acquisition and analysis is temporarily halted. Rows that have not been processed can be edited during the pause. The row currently being processed cannot be edited.

To resume processing, click the Assistant Bar **Pause/Restart** icon or **Pause/Restart** toolbar button, or select **Batch > Pause/Restart** again. Batch processing continues with the row on which it was paused.

3. Stop

To stop batch processing, click the Assistant Bar **Stop** icon or **Stop** toolbar button, or select **Batch > Stop**. Processing stops whether it is currently occurring or it is paused.

4. Edit Method

Click the Assistant Bar **Method Development** icon to edit a method file used by the batch table. The "Data Acquisition" window opens, and the method file, including the instrument parameters, selected in the batch table is displayed. Methods cannot be edited during batch processing.

7.3 Automated Postrun Analysis

This section describes how data processing is automated in the GCMS Postrun Analysis application. For information about the qualitative and quantitative analysis parameters, refer to Chapter 4 "Qualitative Analysis" on page 111 and Chapter 5 "Quantitative Analysis" on page 137.

7.3.1 Creating a Batch Table

1. Open GCMS Postrun Analysis, and click the Assistant Bar **Batch Processing** icon. The "Batch Table" window is displayed.

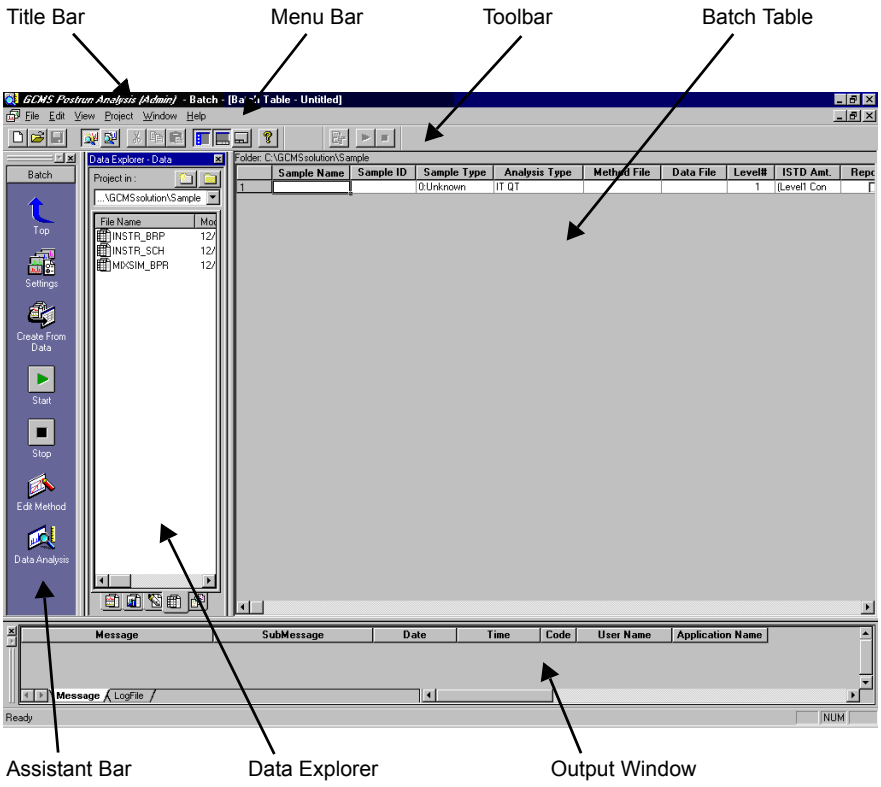


Figure 7.13 "Batch Table" Window

2. Click the Assistant Bar **Settings** icon, and the "Settings" dialog box is displayed.

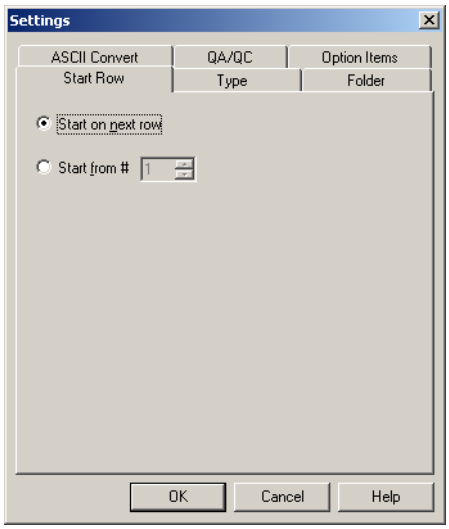


Figure 7.14 "Settings" Dialog Box

For more information about the "Settings" dialog box, refer to the second topic of Section 7.2.1 "Creating a Batch Table".

3. A batch table can be created automatically from data files. Click the Assistant Bar **Select Data Files** icon, and the "Select Data File" dialog box is displayed.

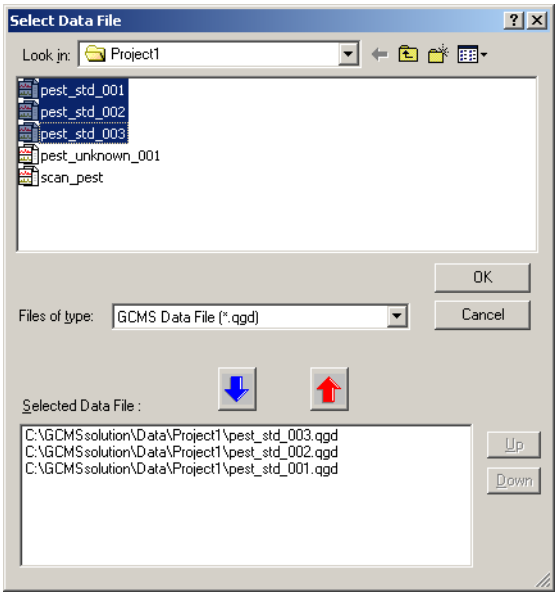


Figure 7.15 "Select Data File" Dialog Box

Select the file to use for data processing. Select multiple files by holding the control or shift keys down while clicking the files with the mouse. Select a file and click the **Down** arrow button to display that file in the text box below. Change the sequence of

the files by selecting a file in the bottom text box and using the **Up** or **Down** arrow button. The batch table is generated with the rows in the displayed order of the files selected. When **Open** is clicked, a batch table is generated from the sample information in the selected data files.

GCMS Postrun Analysis can open and modify batch files created in GCMS Real Time Analysis, however, parameters for data acquisition are not displayed, and only data processing can be performed.

7.3.2 Performing Continuous Postrun Analysis

1. Start
   Click the Assistant Bar **Start** icon or **Start** toolbar button, or select **Batch > Start** to initiate batch processing. Batch analysis starts with the first row or the row indicated in the Settings Start Row tab.

2. Stop
   To stop batch processing, click the Assistant Bar **Stop** icon or **Stop** toolbar button, or select **Batch > Stop**. Processing stops if it is currently occurring.

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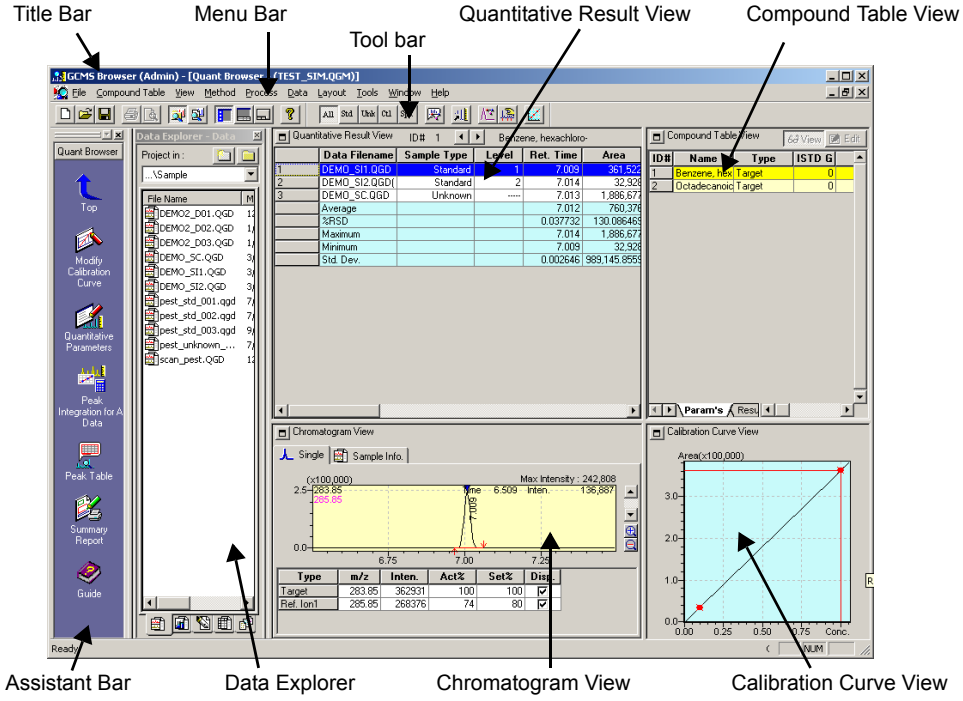
8 Quantitative Browser

8.1 Overview

In Quantitative Browser, the quantitation results of multiple data (concentration, area, height, etc.) are listed for each compound specified in Compound Table in the method file. The listed quantitation results are displayed after being statistically calculated (Average, %RSD, Max, Min, Standard Deviation) as they are needed. You can print out these results as summary reports.



8.1.1 Quantitative Browser Window

Title Bar	Displays currently used application names or process names and method file names.
Menu Bar	Displays various command menus of the displayed window.
Toolbar	Displays buttons for the various command tools for the displayed window.
Assistant Bar	Lists command icons corresponding to the general operation sequence.
Data Explorer	Data file or method file can easily be opened by double-clicking on file icon.
Quantitative Result View	This view loads multiple data files and displays quantitative result (Conc. Area, Height, ...) as well as statistics (average, %RSD, maximum, minimum and standard deviation) in a table format.
Compound Table View	This view displays the Compound Table and Grouping Table of the currently loaded method file, as well as the Identification Result and Grouping Result of the data file selected on the Quantitative Result view.
Chromatogram View	In the data file selected on the Quantitative Result view, the ID chromatogram selected on Compound Table view is displayed.
Calibration Curve View	In the currently loaded method file, the calibration curve of compound which is selected on Compound Table view is displayed.

8.2 The Main Operations in Quantitative Browser Window

Displays the list of the quantitative results of multiple data.	Displays peak integration and quantitative result as well as statistics in a table format. (Reference: 8.2.1 "Displays the List of the Quantitative Results of Multiple Data")
Executes peak integration/quantitation of multiple data simultaneously.	Executes peak integration/quantitation for all compounds of multiple data simultaneously. Moreover changes parameters for peak integration or identification or modifies the calibration curve. (Reference: 8.2.2 "Execute Peak Integration/Quantitation of Multiple Data Simultaneously")
Outputs the list of quantitation results as summary report.	Outputs the list of quantitative results and statistics as summary report. (Reference: 8.2.3 "Output the List of Quantitation Results as Summary Report")

8.2.1 Displays the List of the Quantitative Results of Multiple Data

The Quantitative Browser displays the quantitative results of multiple data which are calculated based on one single compound table. The compound table is loaded from the method file you directly specified, or from the method file used for the quantitation of the data file you loaded on Quantitative Browser.

1. To load a method file

(1) Click on the Method tab in Data Explorer. Data Explorer becomes the method file mode.

(2) Double-click the icon of the method file to be loaded. All data files which constitute the calibration curves (Sample type: Standard) included in the method file are loaded automatically.

2. To load a data file

(1) Click on the Data tab of the Data Explorer. Data Explorer becomes the data file mode.

(2) Double-click on the appropriate data file.

3. To load a batch file

(1) Click on the Batch tab of Data Explorer. Data Explorer becomes the batch file mode.

(2) Drag and drop the appropriate batch file to the Quantitative Browser. The Quantitative Result view searches for the row for which sample type is "Standard" in the batch file, and loads the method file for the first hit row and all data files which use the method file. If no such row is found on the batch file, the Quantitative Result view searches for the "Unknown" row, and loads the method file on the first hit row and all data files which use the method file.

4. To switch data type
In Quantitative Result view, you can extract and display its loaded data by their sample type, such as [Standard], [Unknown], [Control] and [Spiked]. You can also display [All] data despite of their sample type. These can be switched on the [Data Type] on the [View] menu or by pushing the buttons on the toolbar.

Note

[Standard] here means the data that constitutes the calibration curve in a method file. Even if the sample type of a data is actually standard, it is treated as [Unknown] if it does not constitute the calibration curve in the opened method file.

5. To switch compounds for the list of quantitative results
On Quantitative Result view, the quantitative results are displayed for each compound on Compound Table view. To switch, select the desired compound on the ID spin button on the upper part of Quantitative Result view, or select the row of the compound on Compound Table view.

Note

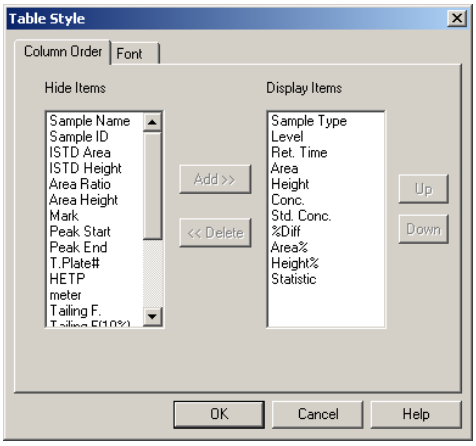
For the Standard sample on Quantitative Result view, you can display the difference between calculated concentration and set concentration on the same level on Compound Table.

Note

The calibration curve of target compound is displayed on Calibration Curve view.

6. To sort all lines in the Quantitative Result view
Select the **View** > **Quantitative Result** > **Sort by Filename** menu, to sort all lines in the Quantitative Result view by filename.

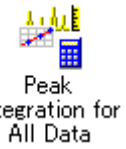
7. To show/hide items on Quantitative Result view, or change the order
Select the **View > Quantitative Result > Table Style** command, to set the parameter in "Table Style" window.



8. Confirm the statistics
Statistics (average, %RSD, maximum, minimum, and standard deviation) are computed and displayed based on the data that is displayed in this view. You can execute statistic calculations for all cells other than data file name, sample name, sample ID, sample type, set concentration, level#, mark, and statistics.

8.2.2 Execute Peak Integration/Quantitation of Multiple Data Simultaneously

1. If you click on the **Peak Integration for All Data** in Assistant Bar, peak integration and quantitation for all compound IDs in all data files are executed.



2. To analyze only data file selected on Quantitative Result view, select the **Process > Peak Integration > Peak Integrate for all IDs** command.

3. To analyze only displayed ID in the selected data file on Quantitative Result view, select the **Process > Peak Integration > Peak Integrate by ID** command.

8.2.3 Output the List of Quantitation Results as Summary Report

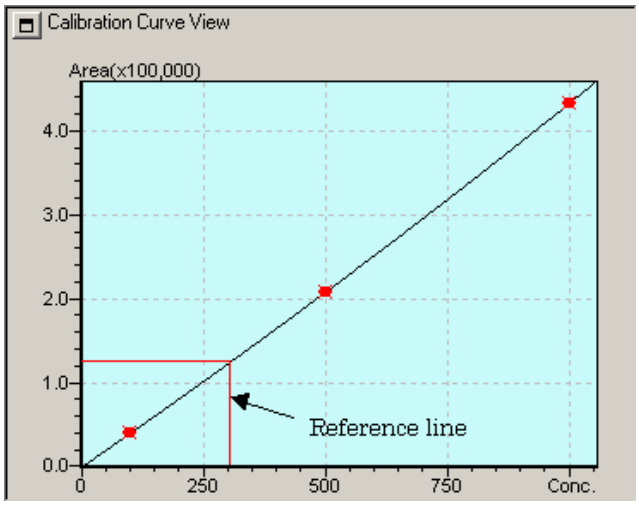
1. Click on the **Summary Report** icon in Assistant Bar, then Report Editor window appears. (A report format has already been loaded.)

2. To change the report format, double-click on the Summary (compound) item and edit on Summary (Compound) Properties window. When **File > Save Format File As** command is selected, your change is saved in the file named as "MS Report Quantitative Browser Report.qgr", and will be used for the next summary output.

3. If there is no need to change the format, click on the **Print** icon in Assistant Bar. The report will be printed out.

8.3 Verify the Calibration Curve

The calibration curve view displays the calibration curve of the compound selected by the Param's or Results tab in the compound table. When the Group Param's or Group Results tab is displayed on the compound table view, only group calibration curves are displayed.

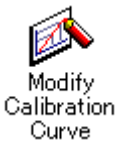


Calibration points and points before averaging are displayed on the calibration curve view. Also on calibration curve, the reference lines are displayed to indicate the area of the peak identified in the date file selected in Quantitative Result view, and the calculated concentration. By these lines, you can easily verify which part of the calibration curve corresponds to the quantitation result.

1. Modify the calibration curve

Click on the **Modify Calibration Curve** icon in Assistant Bar, "Calibration Curve" window opens.

The method file which is loaded in the "Quantitation Browser" window is automatically loaded.



REFERENCE

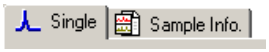
To add/delete the data file of standard sample to/from calibration point, refer to "5.3 Creating Calibration Curves".

Note

When you return to "Quantitative Browser" window from the "Calibration Curve" window, the browser automatically re-executes the quantitation for compound ID of all data files whose calibration curve were modified.

8.4 Manual Peak Integration and Manual Peak Identification

The chromatogram view consists of the Single tab and the Sample Info. tab. Both tabs display the data file information selected on the quantitative result table.



On Single tab, chromatogram of selected compound on Compound table is displayed, and you can verify whether the target peak is detected or properly identified. The chromatograms (Max: 5) of the reference ions are overlaid with the target chromatogram. In addition, the table of the reference ions information is displayed below the chromatogram. For these chromatograms, you can manually specify peaks (Manual Peak Integration,) or change peaks to be identified (Manual Identification).

1. Manual Peak Integration

(1) Select the **Process > Peak Integration > Manual Peak Integrate** command. The cursor on the chromatogram changes to a vertical line, select the time range for the peak to use by dragging with the mouse. The select Base Line dialog box is displayed, and select how the base line should be drawn.

Note

When manual peak integration is performed, the peak identification will also be performed automatically.

2. Manual Identification

(1) Select the **Process > Manual Identification** command. The cursor on the chromatogram changes to a vertical line, click in the peak area at the retention time to identify. The peak will then be identified. If the wrong peak has been identified, the identification can be changed by dragging the identification mark displayed on the peak in the chromatogram to the correct peak to be identified.

8.5 Save the Layout of Quantitative Browser Window

You can save, loaded method file name, data file name, the information of selective (highlighted) data file /compound on Quantitative Result View/Compound Table View, as Browsing File (extension: *.qgq). By reloading this browsing file, you can easily restore the status of these files.

1. Select the **Layout > Save Browsing File As** command.

REFERENCE

For the using "Save File As" dialog box, refer to "2.9.2 Opening and Saving Files".

Note

The position of the view, the size, the color information, etc. are saved for each user who can log on, instead of this browsing file.

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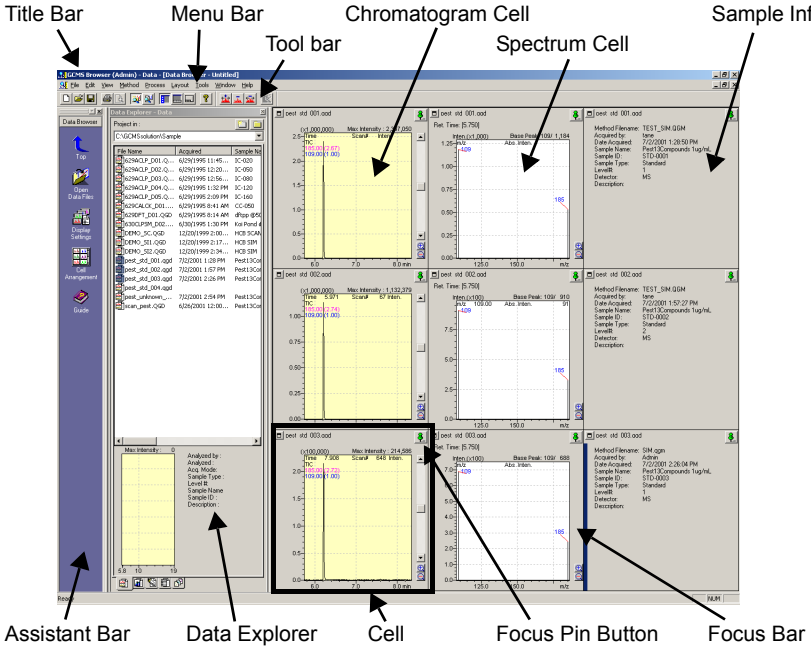
9 Data Browser

9.1 Overview

In the Data Browser, the chromatograms and spectra in multiple data files can be displayed simultaneously. The Data Browser window consists of the cells to display information. Each cell can display the chromatogram or spectrum loaded from a single data file, and by increasing cells, the information on a maximum of 64 cells are displayed simultaneously (8 cells each in vertical and horizontal). By operating a cell, (zoom, changing retention time, etc.) you can co-execute the operation for chromatogram or spectrum on each of the cells. By removing focus pins, you can operate cells independently.

9.1.1 Data Browser Window



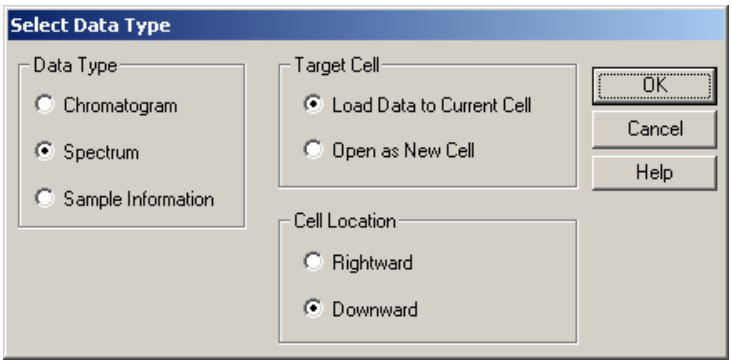
Title Bar	Displays the currently used application name, or process name and data file name.
Menu Bar	Displays various command menus of the displayed window.
Toolbar	Displays buttons for the various command tools for the displayed window.
Assistant Bar	Lists command icons corresponding to the general operation sequence.
Data Explorer	Data file can easily be opened by double-clicking on file icon.
Chromatogram Cell	Displays the chromatogram and its information with the specified group and mass (TIC, MIC, MC).
Spectrum Cell	Displays the spectrum and its information at a specified retention time.
Sample Information Cell	Displays sample information (Method Filename, Acquired by, Date Acquired, etc.) of data file.
Cell	A cell can load one single data file. In Data Browser window, the information on a maximum of 64 cells are displayed simultaneously (8 cells each in vertical and horizontal).
Focus Pin Button	If the pin of the cell is pressed (indicated in green), the cell receives the information sent from other cell.

Focus Bar	This is displayed for focused cell. Menu commands are valid for cells with the focus bar.

9.1.2 Opens the Data File

1. Click on the Data tab in Data Explorer.

2. Double click the desired data.
The "Select Data Type" window is opened.



3. Select the data type and the target cells.

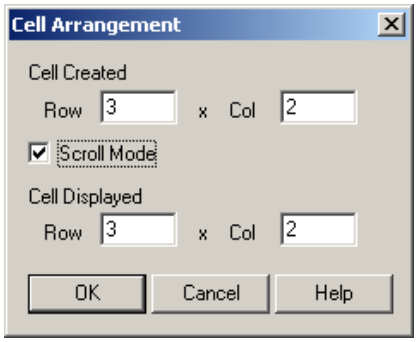
Parameter	Description
Data Type	Chromatogram: Displays the chromatogram and chromatogram information. Spectrum: Displays the spectrum and spectrum information. Sample Information: Displays the sample information.
Target Cell	Load Data to Current Cell: Loads into the currently selected cell. Open as New Cell: Adds a new cell.
Cell Location	Rightward: Adds a cell for a column and displays. When Open as New Cell is selected, the browser adds the same number of columns as data files exist, and then loads the data files on the first row. Downward: Adds a cell for a row and displays. When Open as New Cell is selected, the browser adds the same number of rows as data files exist, and then loads the data files on the first column.

4. The Data file is loaded in the specified cell.

9.1.3 Changes the Cell Layout

Specify the number of cells displayed on the window.

1. Click on the **Cell Arrangement** icon in Assistant Bar.
The "Cell Arrangement" window is opened.



2. The following setup is carried out.

Parameter	Description
Cell Created	Divides the data browser into the number of cells specified here in a grid shape. Row: Specifies the number of rows. Col.: Specifies the number of columns.
Scroll Mode	When checked, the horizontal or vertical scroll bar is displayed as necessary.
Cell Displayed	Specify the number of rows and columns displayed on the data browser at one time. Valid only when Scroll Mode is selected.

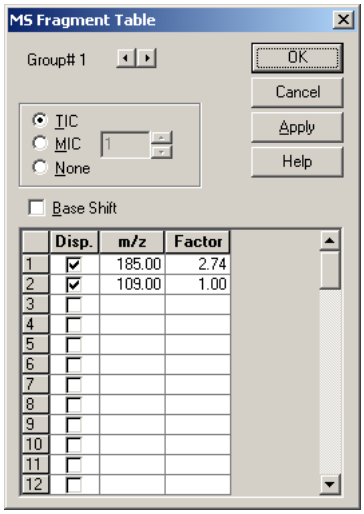
9.2 Operation on Chromatogram Cell

This window displays the chromatogram with the specified group and mass (TIC, MIC, MC). The displayed chromatograms are determined on the fragment table. Chromatograms are either overlaid or in the stack display. When analysis is being performed with multiple groups (SIM ion sets), only the chromatogram for one of the groups is displayed. To change the group is performed by Next Group command or Previous Group command from View menu.

9.2.1 Sets the Displayed Chromatograms

The displayed chromatograms in Chromatogram cell are determined on the Fragment Table window.

1. Select the **View > Fragment Table** command, the "MS Fragment Table" is displayed.



2. In the MS Fragment Table window, choose between displaying TIC, MIC, or None in the chromatogram window. In addition, specify MC m/z and magnification factors. When displaying the MIC chromatogram, MIC table needs to be set up beforehand. To edit the MIC table, click the **Method > Data Load Format** command.

9.2.2 To Work with Other Cells

If you click a focus pin button, the pin is stuck (indicated in green), and by operating the cell, you can co-execute the similar operation for the other pin-stuck cells.

1. If you zoom a chromatogram, the chromatograms on the other chromatogram cell are also enlarged in the same Time and Intensity range.

2. If you double click on chromatogram, the spectrum corresponding to the retention time is displayed on spectrum cell.

3. If you execute a spectrum calculation on chromatogram, the spectrum calculation within the same time range is also performed on the other chromatogram cells, and the calculated spectrum is displayed on each of the spectrum cells which has loaded the same data file.

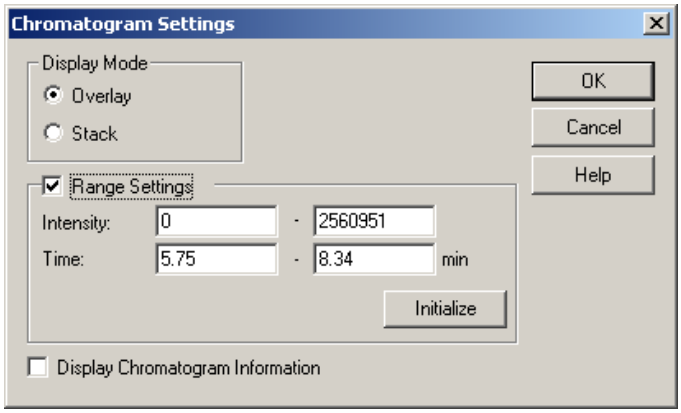
Note

If the retention time is in FASST measurement interval, the Scan spectrum is displayed.

9.2.3 To Change Display Range

1. To set the display range of chromatogram, drag on the chromatogram or click on the **Display Settings** icon in Assistant Bar.

2. If "Chromatogram Settings" window is opened, carry out the following setup.



Parameter	Description
Display Mode	Select Overlay or Stack.
Range Settings	When the Range Settings is checked, the range of the intensity axis and the time base will be specified. When the check is OFF, the chromatogram from the starting time of the analysis to the end on the current group will be displayed. Click on the Initialize button to normalize the X-axis and Y-axis.
Display Chromatogram Information	Select whether to display the chromatogram information.

9.2.4 Print the Cell Image

1. To print a report, select the **File > Print Image for Selected Cell** command, the Report Editor window appears. (A report format has already been loaded.)

2. To change the report format, double-click items and edit on the property window. When **File > Save Format File As** command is selected, your change is saved in the report format file named as "Data Browser MS Chromatogram Report.qgr", and will be used for the next image printing of chromatogram cell.

3. If you have no need to change the format, just click on the **Print** icon in Assistant Bar. The report will be printed out.


Print

9.3 Operation on Spectrum Cell

This window displays the spectrum at a specified retention time.

9.3.1 To Display Spectrum within Specified Retention Time

1. Double-click on chromatogram cell at a retention time. In this case, focus pins need to be stuck both on Chromatogram cell and Spectrum cell.

9.3.2 Subtracting Spectrum

1. Stick focus pin on Spectrum cell, and click on the **Subtract Spectrum** icon in tool bar. 

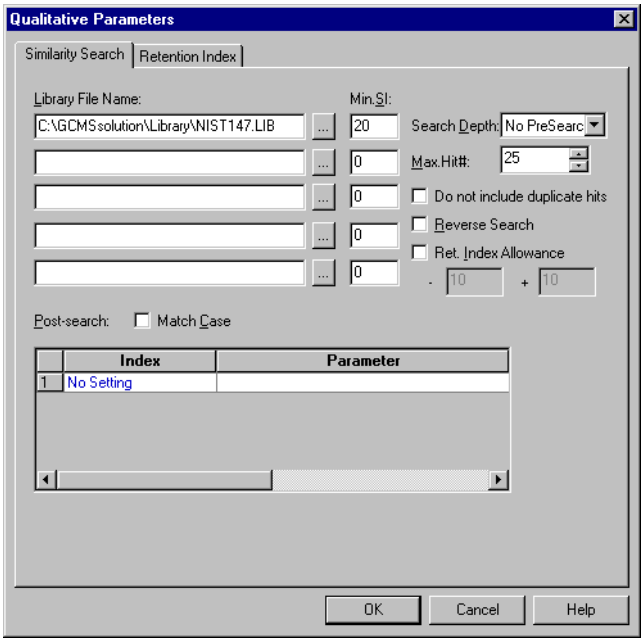
2. Stick focus pin on chromatogram cell, and double-click on the chromatogram on chromatogram cell to specify the retention time you need. Then the spectrum is subtracted, and the result is displayed on spectrum cell.

REFERENCE

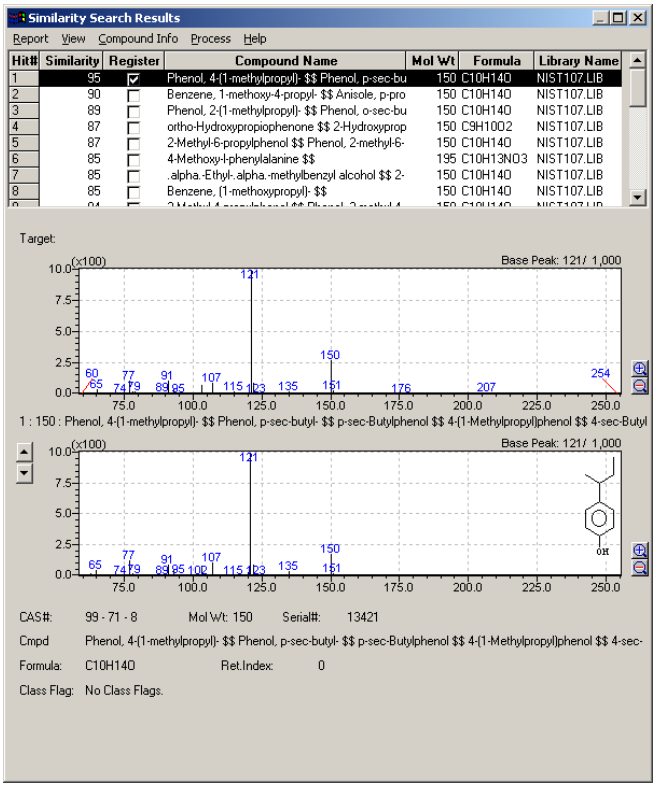
About the averaging the spectrum or subtract averaged spectrum, refer to "4.2.2 Averaging the Spectrum and Background Processing".

9.3.3 To Execute Similarity Search for Spectrum

1. Select the **Process > Similarity Search for Current Spectrum** command.
   If the library file name is not specified to use for similarity search, set on the Similarity Search tab in Qualitative Parameters and select the **Process > Similarity Search for Current Spectrum** command.



2. The "Similarity Search Results" window will be open.



9.3.4 To Work with Other Cells

If you click a focus pin button, the pin is stuck (indicated in green,) and by operating the cell, you can co-execute the similar operation for the other pin-stuck cells.

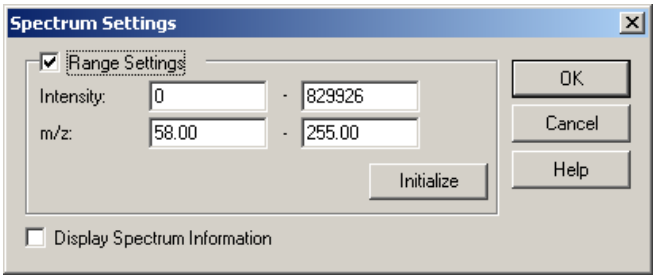
1. If you enlarge a spectrum, the spectra on the other spectrum cells are enlarged in the same m/z and Intensity range.

2. If you double-click on spectrum, the mass chromatogram of the m/z is displayed on chromatogram cell of scan data.

9.3.5 To Change Display Range

1. To set the display range of spectrum, drag on the spectrum or click the **Display Settings** icon on Assistant Bar.

2. If "Spectrum Settings" window is opened, carry out the following setup.



Parameter	Description
Range Settings	When the Range Settings is checked, the range of the intensity axis and the m/z axis will be specified. When the check is OFF, the spectrum from the starting m/z of the analysis to the end on the current group will be displayed. Click on the Initialize button to normalize the X-axis and Y-axis.
Display Spectrum Information	Select whether to display the spectrum information.

9.3.6 To Print the Image of a Cell

1. To print a report, select the **File > Print Image for Selected Cell** command, then Report Editor window appears. (A report format has already been loaded.)
2. To change the report format, double-click items and edit on the property window. When **File > Save Format File As** command is selected, your change is saved in the report format file named as "Data Browser MS spectrum Report.qgr", and will be used for the next image printing of spectrum cell.
3. If you have no need to change the format, just click on the **Print** icon in Assistant Bar. The report will be printed out.

9.4 Save the layout in Data Browser Window

On Data Browser, you can save the information of each cell such as data file name, data type, cell position, etc. as layout file (*.lyt). By opening this layout file, you can easily reload the series of data files relating to each of the cells.

1. Select the **Layout > Save Layout File As** command.

REFERENCE

For the using "Save File As" dialog box, refer to "2.9.2 Opening and Saving Files".

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10 Maintenance

10.1 Precautions

Read and follow the following MS maintenance precautions to ensure optimal MS performance and prevent accidents.
Shut down all modules including the MS before performing GC maintenance as described in the GC user manual.

WARNING

1. Follow all relevant precautions when performing a maintenance operation.
2. Wear clean gloves when working on the ion source box or interior of the mass spectrometer.
3. Use clean tools to disassemble parts. Contaminants from dirty tools or gloves can generate background noise. Clean any tools used inside the mass spectrometer with a lint-free cloth and acetone.
4. Place parts on a clean cloth as they are removed, to ensure that they remain clean.
5. Take note of how the parts are assembled to ensure correct re-assembly.
6. Operations that require opening the power unit or moving the MS should be performed by a Shimadzu representative. Only changing columns and minor ion source box maintenance should be performed by the user.

10.2 Filament Replacement

The filament can burn out after extended use or by a decrease in vacuum level. This section describes changing the filament. Follow these precautions when performing this procedure.

WARNING

Electric shock hazard. Turn off the instrument and confirm that the main switch is off. Confirm that the circuit breaker for the instrument is off.

WARNING

The ion source and interface are usually heated during operation. Before maintenance, allow the instrument to cool for at least 10 minutes once it has been turned off.

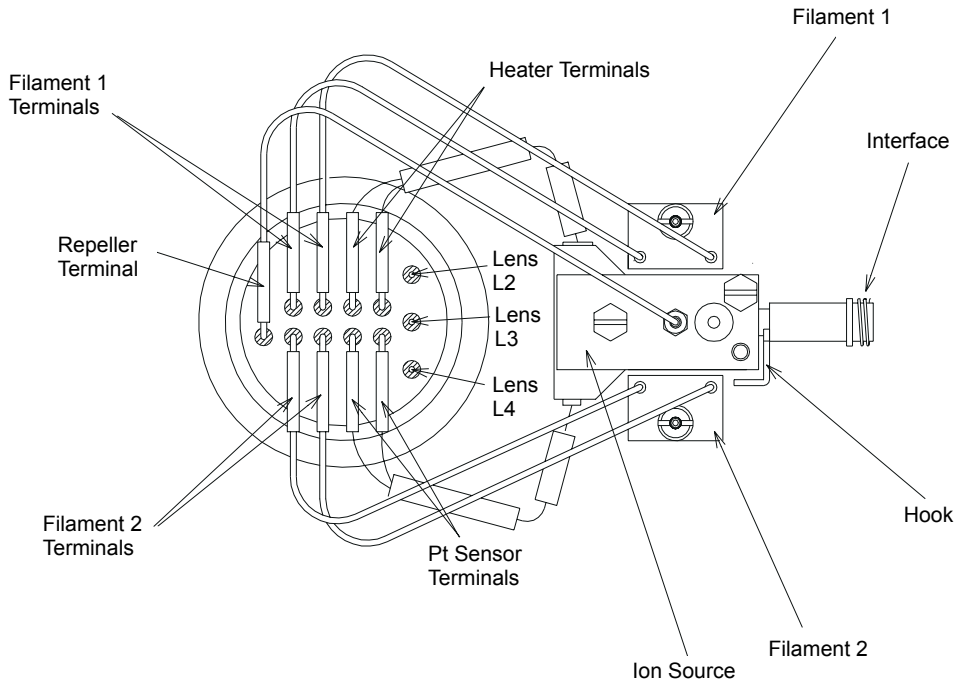


Figure 10.1 Ion Source Box Diagram

10.2.1 Filament Replacement Procedure

1. Turn off the instrument as described in Section 2.4.2 "Shutting Down the Instrument", page 26.
2. Confirm that the main instrument switch is off.
3. Confirm that the circuit breaker for the instrument is off.
4. Rotate the knob and open the front panel.
5. Disconnect the filament by pulling the lead connectors from the lead pins (1).
6. Loosen the retaining nut (2) and pull out the filament (3).

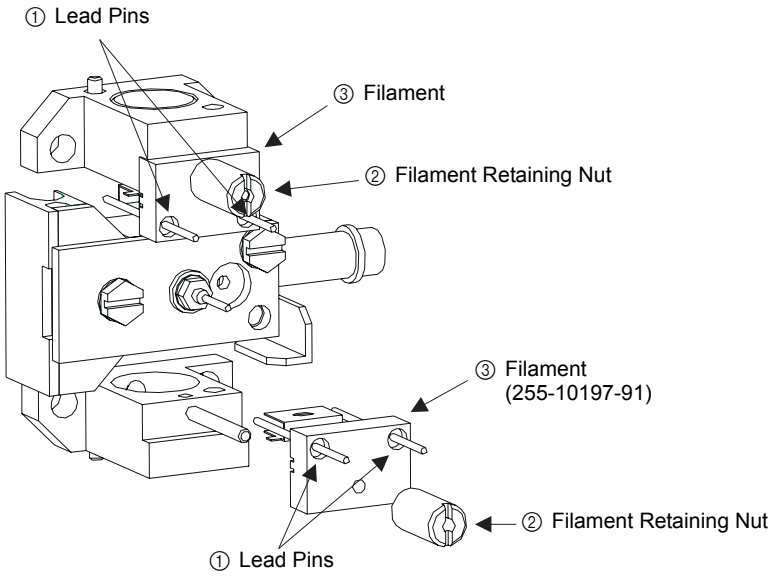


Figure 10.2 Ion Source

7. Completely insert the new filaments and tighten the nut.
8. Reconnect the filament leads by pushing the lead connectors onto the lead pins.

Note

Precautions during filament installation:
1. Insert the filament completely.
2. Do not allow the filament leads to contact any other part when the filament is seated.

10.3 Cleaning the Ion Source Box and Repeller Electrode

The function of the ion source is to bombard a sample with electrons, causing ionization and therefore the production of ions. Consequently, the source parts become contaminated over time. When the ion source box or repeller electrode are contaminated, the generated ions are not produced effectively, decreasing sensitivity. Remove and clean the ion source box periodically.

WARNING

Electrical shock hazard. Turn off the instrument and confirm that the main instrument switch and circuit breaker are off.

WARNING

Exercise caution when handling the organic solvent used to clean the ion source box. Provide proper ventilation in areas where organic solvent is used.

WARNING

The ion source and interface are usually heated during operation. Before maintenance, allow the instrument to cool for at least 10 minutes once it has been turned off.

WARNING

There is a risk of burns because the ion source box and repeller electrode are still hot after drying/baking. Make sure that parts cool sufficiently before maintenance.

10.3.1 Removing the Ion Source Box and Detaching the Repeller Electrode

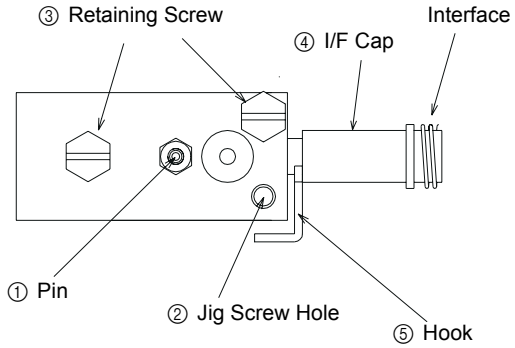


Figure 10.3 Ion Source Box Diagram after the Filaments Have Been Removed

1. Shut down the instrument according to Section 2.4.2 "Shutting Down the Instrument", page 26.

2. Confirm that the main instrument switch is off.

3. Confirm that the instrument circuit breaker is off.

4. Rotate the knob and open the front panel.

5. Carefully pull the lead connector from the pin of the repeller electrode in the figure above with forceps.

6. Screw the jig for attaching/detaching the ion source box into the threaded hole on the box.

7. Loosen the retaining screws (3) that supports the ion source box by a half turn.

8. Push the interface cap (4) to the right, and rotate the hook (5) downward.

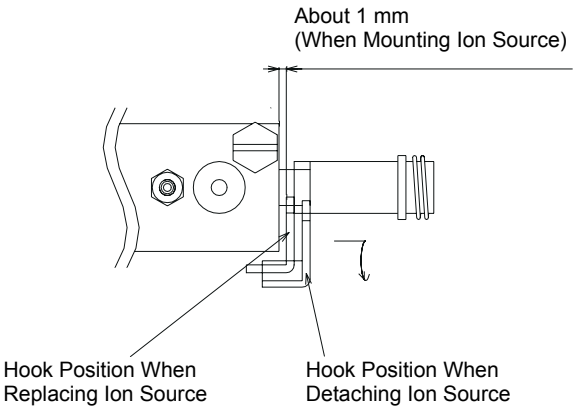


Figure 10.4 Positioning the Hook

9. Loosen the retaining screws (3) and detach the box.

10. Detach the jig and separate the box and repeller.

11. Loosen the nut with the wrench and detach the repeller electrode.

12. Clean according to Section 10.3.3 "Cleaning the Ion Source Box and Repeller Electrode", page 223.

10.3.2 Re-Assembling the Ion Source Box and Repeller Electrode

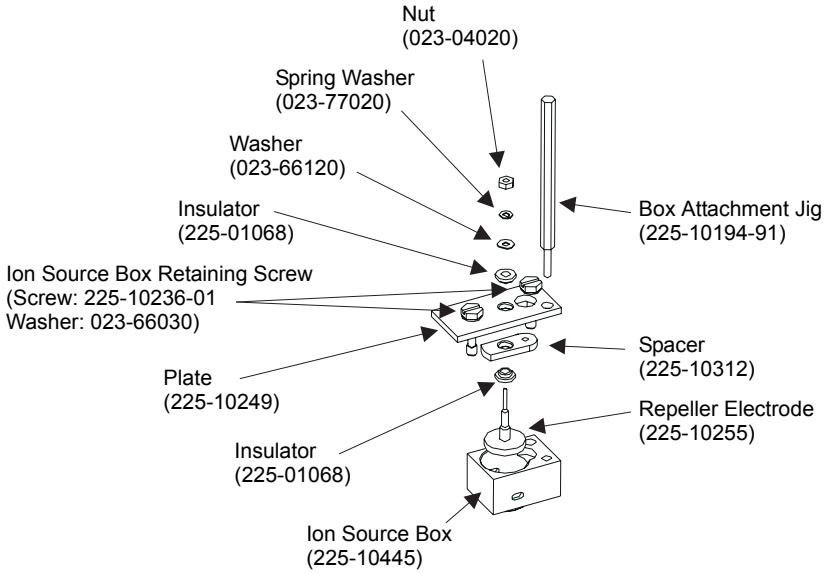


Figure 10.5 Ion Source Box and Attachment Jig

1. Place the repeller electrode in the ion source box and adjust so that the repeller is positioned in the center of the ion source box.

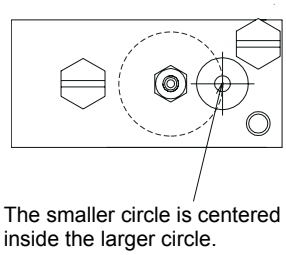


Figure 10.6 Positioning the Repeller Electrode

2. Install the parts in the ion source box as shown in the diagram. (Figure 10.5 "Ion Source Box and Attachment Jig") Use the two retaining screws to temporarily hold the parts in place.

3. Attach the installation jig to the ion source box.

4. Push up the hook on the interface cap, and attach it onto the box.

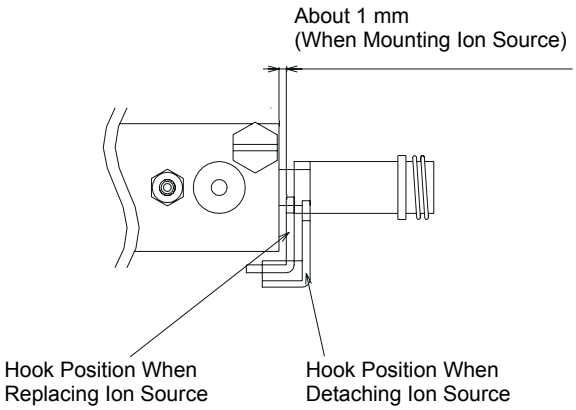


Figure 10.7 Positioning the Hook

5. Adjust the jig vertically so that the interface cap is completely inserted into the box.

6. Detach the jig and tighten the two retaining screws used to hold the parts in place. The left screw should be tightened first.

7. Attach the repeller lead connector.

Note

1. Verify that the cap at the end of the interface is seated firmly in the hole on the ion source.
2. Ensure that the repeller electrode lead connector is attached.
3. Verify that the ion source is seated straight.

Note

Precautions for reassembly of the ion source box and lens 1.

1. Do not use excessive force to tighten the screws; this could bend the lens or break the insulating parts.
2. Use a circuit tester to ensure that the repeller electrode and the ion source box are insulated.
3. Do not clean the insulator parts. New parts can be purchased if the parts are contaminated.

10.3.3 Cleaning the Ion Source Box and Repeller Electrode

1. Polish parts with abrasive paper. Refer to Section 10.3.4 "Required Materials", page 223.

Note

Precautions for polishing the ion source box and repeller electrode:

1. Be careful not to bend the electrode when polishing it.
2. Remove dust from the surface with a clean compressed air source.
3. Perform ultrasonic cleaning in acetone or petroleum ether.
4. Dry for about 30 min at 90 - 120 °C. When analyzing trace amounts or easily adsorbed compounds, bake at 400 °C for an hour in a commercially available furnace.
5. Store the dry ion source box and repeller in a clean box.

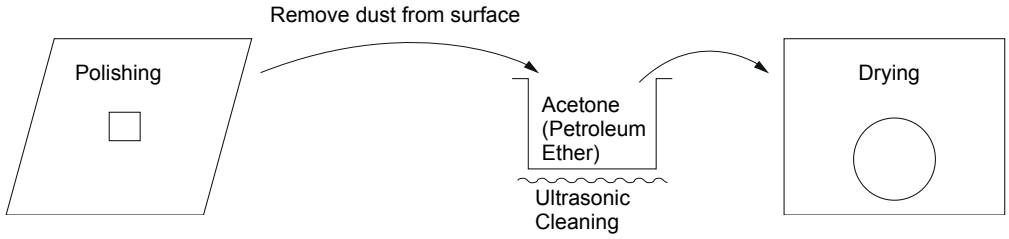


Figure 10.8 Cleaning the Ion Source Box and Repeller Electrode

10.3.4 Required Materials

1. Abrasive paper

- P/N 085-35124-02 (general finishing, 20 sheets)
- P/N 085-35124-03 (extra-fine finishing, 20 sheets)

If parts are extremely contaminated, polish with general finishing paper followed by extra-fine finishing paper. If parts are only slightly contaminated, polish with extra-fine finishing paper only.

10.4 Vacuum Pump Maintenance

This section describes how to change the rotary pump oil and replace the lubricant for the main turbomolecular pump. The rotary pump oil is still hot after shutting down the system. Wait at least 10 minutes before performing maintenance.

10.4.1 Changing the Rotary Pump Oil

The rotary pump (model E2M1.5) requires an oil change every 3000 hours. The oil must be changed to prevent vacuum level deterioration, oil leaks and excessive noise.

The oil must have the proper characteristics for the pump. Use only pure oil of the following specification: Rotary pump oil Ultragrade 15 1L (P/N 017-30163-11).

Oil Change Procedure

WARNING

Danger of serious burns. The rotary pump oil is hot immediately after shutdown. Wait at least 10 minutes before performing maintenance.

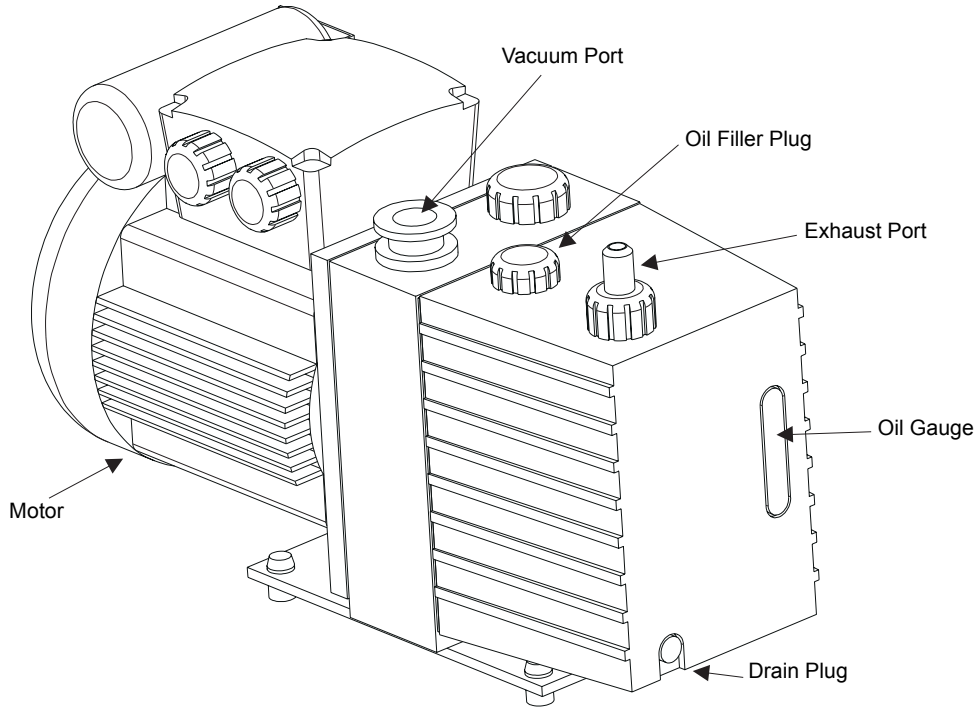


Figure 10.9 Rotary Pump

1. Shut down the instrument according to "Shutting Down the Instrument" in Section 2.4.2 "Shutting Down the Instrument", page 26.
2. Wait at least 10 min.
3. Turn the rotary pump motor switch off.
4. While using an appropriate container and wearing rubber gloves remove the drain plug and drain the oil. Use caution: Oil will be discharged when the drain plug is removed.
5. When the oil has finished draining, replace the drain plug temporarily and turn the motor switch on.
6. Click the Assistant Bar **Vacuum Control** icon to open the "Vacuum Control" dialog box.

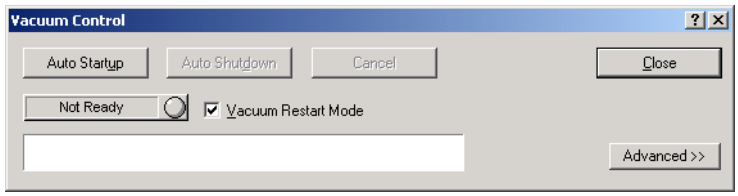


Figure 10.10 "Vacuum Control" Dialog Box

7. Click the **Advanced** button in the lower part of the window to access the manual operation system. Click the **Start** button of the rotary pump adjacent to the Rotary Pump oil replacement item in the Vacuum System group.

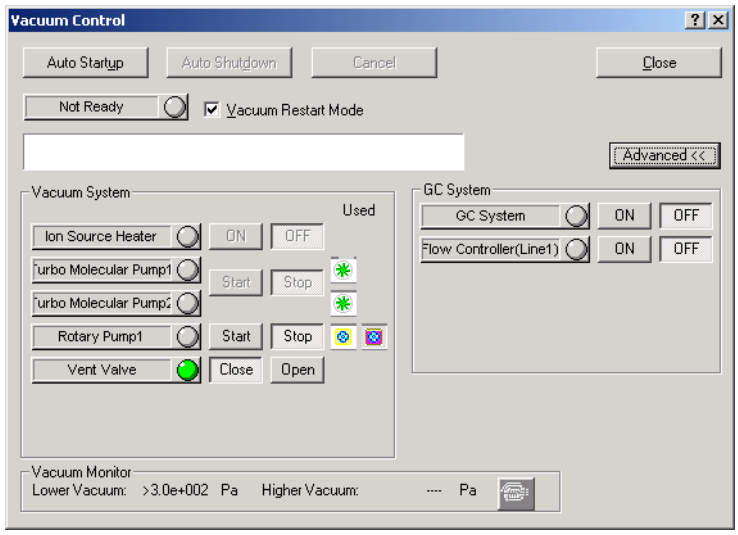


Figure 10.11 "Advanced Vacuum Control" Dialog Box

8. Leave the rotary pump on for 5 - 10 sec. The pump will operate, discharging the remaining oil inside the pump.
9. Click the rotary pump **Stop** button in the "Vacuum Control" dialog box.

10. Turn the rotary pump motor switch off.
11. Remove the drain plug, and drain the remaining oil.
12. Close the drain plug, and remove the oil filler plug. Add fresh oil until the oil reaches the MAX mark on the oil gauge (about 0.28 L).
13. Replace the oil filler plug.

Note

In addition to the oil changing procedure, pump overhaul should be performed every 1500 hours. Refer to the E2M1.5 user's manual.

10.4.2 Changing the Lubricant for the Turbomolecular Pump

The turbomolecular pumps installed in some GCMS-QP2010 models need to have the lubricant replaced. The QP2010 models for which replacement of the lubricant is necessary are shown in the following table (If the standard turbomolecular pump provided in the QP2010 has been replaced with that of another model, this may make replacement of the lubricant necessary in some cases even if the QP2010 model is not listed as an applicable model in the table). Please contact your service representative when you need to replace the lubricant.

Frequency of replacement of lubricant	Parts number of QP2010
Not necessary	225-10000-xx, 225-10001-xx, 225-10002-xx 225-10005-xx, 225-10006-xx, 225-10007-xx 225-10040-xx, 225-10041-xx, 225-10042-xx 225-10085-xx, 225-10086-xx, 225-10087-xx 225-10003-xx
Every 1 to 1.5 year	225-10080-xx, 225-10081-xx, 225-10082-xx 225-10090-xx, 225-10091-xx, 225-10092-xx

xx=24, 34, 37, 39, 91 or 92

Note

It is recommended that turbomolecular pump overhaul is performed every 25000 hours.

10.5 Cleaning the Fan

The fan and fan guard must be periodically cleaned.

> **Note**
>
> Do not use the instrument if dust has accumulated on the fan or fan guard. Insufficient cooling could compromise performance.

10.5.1 Cleaning the Fan

1. Shut down the instrument according to the procedure described in Section 2.4.2 "Shutting Down the Instrument", page 26. Ensure that all of the power switches are off.
2. Vacuum dust from the vents of the MS.
3. Vacuum dust from the fan and fan guard on the back of the gas chromatograph (GC).
4. Vacuum dust from the vents of the personal computer (PC) system and any other instrument modules, such as the autosampler.

> **WARNING**
>
> To prevent injuries or accidents when cleaning the fan and fan guard, ensure that instrument is shut down and verify that all power switches are off.

10.6 Checking for Leaks

This section describes how to check the instrument for leaks. If an air leak is clearly present, locate the leak as described in Section 10.6.2 "MS Vacuum Leak Check", page 232, and take corrective measures.

Operating an instrument with an air leak can cause a decrease in sensitivity, an increase in noise, filament burnout or other problems. Always check for leaks after performing maintenance.

A GC leak will have different symptoms than an MS leak. Normally, carrier gas will leak from the system if there is a GC leak, while air will be drawn into the system when there is an MS leak. To check for GC leaks, refer to Section 10.6.1 "GC Carrier Gas Leak Check", page 228. To check for MS leaks, refer to Section 10.6.2 "MS Vacuum Leak Check", page 232 and Section 10.6.3 "Vacuum Leak Check Using Peak Monitor", page 233.

10.6.1 GC Carrier Gas Leak Check

To check for GC carrier gas leaks, a blank plug (vespel ferrule filled with wire) is installed in the injection port column connection, and the split and purge vents are sealed with a blank nut. This seals the carrier gas in the injection port flow line, the carrier gas flow line, and the split and purge gas flow lines. Leaks are detected by observing the change in pressure of the sealed carrier gas over time. Changes in carrier gas pressure are observed by monitoring flow from the GC-2010 Keypad and Display Panel.

1. Shut down the instrument according to Section 2.4.2 "Shutting Down the Instrument", page 26. Turn the MS power source off, but leave the GC-2010 power source on.
2. Open the GC oven, and remove the column on the injection side.
3. Install the blank plug in place of the column in the injection port.
4. Install G-type blank nuts in the vents for the split and purge gas, respectively.

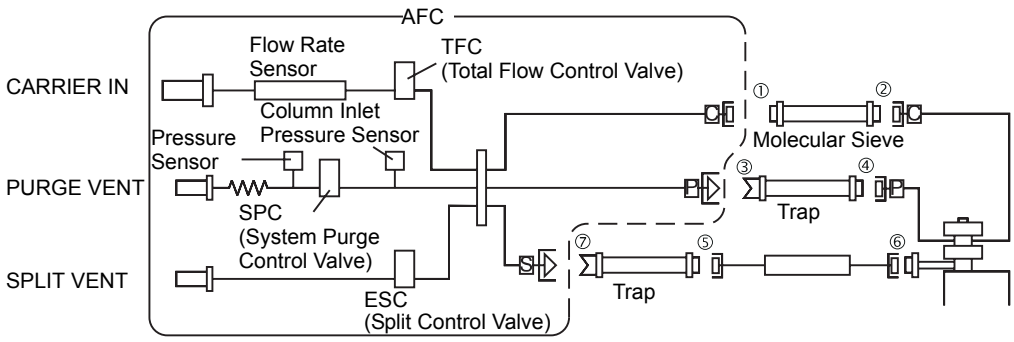


Figure 10.12 Flow Path Diagram

5. Ensure that the carrier gas supply pressure (cylinder pressure) exceeds 350 kPa.
6. On the GC-2010 "Flow key" screen, set the Injection mode to "Direct" and the Control mode to "Pressure."
7. Set the injection pressure at 350 kPa, press ON/OFF (PF Menu) and turn on the AFC.

8. Wait 5 minutes and verify that the injection pressure is between 300 and 400 kPa. If the pressure is above 400 kPa, loosen the column nut slightly and let the pressure decrease. If the pressure is below 300 kPa, increase the injection pressure slightly.
9. Verify that the total flow is below 2 mL/min. A total flow greater than 2 mL/min indicates a carrier gas leak.
10. Press ON/OFF (PF Menu) on the "Flow key" screen and turn off the AF Control.
11. Allow pressure to stabilize after turning off the AF Control.
12. Verify that the pressure does not decrease by more than 30 kPa per hour. A pressure decrease exceeding 30 kPa per hour indicates a leak.
13. While the carrier gas is sealed under pressure, possible leaks can be checked by using an electronic gas leak detector (available commercially) at the connections for each flow path, such as the G-type nuts and injection port nuts.
14. When a leak is detected, tighten the part or remove it and inspect for problems. Replace the three aluminum spacers when a G-type nut is removed.

15. If a leak was detected because of a pressure decrease after step 14, replace the injection port septum, and verify that the O-ring is properly installed on the glass insert in the injection port. Repeat the pressure leak test described in steps 8 - 12.

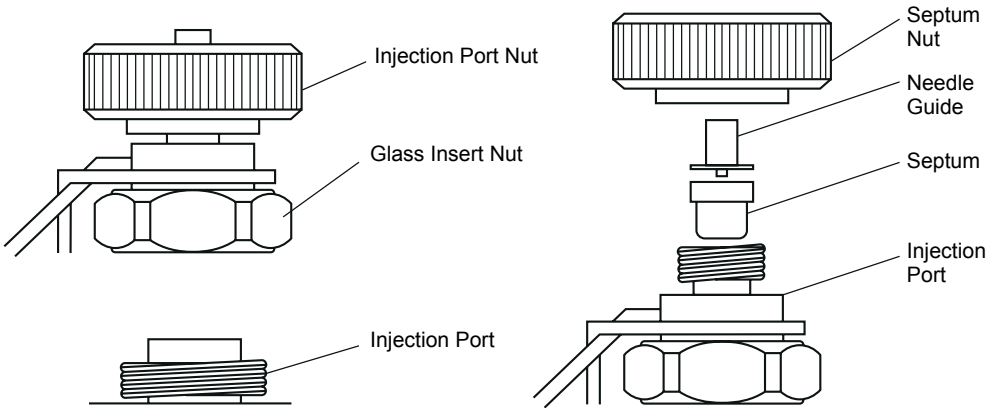


Figure 10.13 Top of Injection Port Assembly

16. If a leak is detected after step 15, tighten the nuts of the injection port assembly, and repeat the pressure leak test described in steps 8 - 12.

17. If a leak continues to be detected, the leak is probably located in the flow lines of the carrier gas, split or purge vents. Tighten connections (1) - (7) in the flow path diagrams shown in Section 10.6.1 "GC Carrier Gas Leak Check", page 228, or remove the parts and check for problems. Replace the three aluminum spacers when a G-type nut is removed.

18. Repeat the pressure leak test described in steps 8 - 12.

Note

Precautions for sealing GC carrier gas under pressure:

1. Ensure that there are no leaks after disassembling and reconnecting a G-type or M-type nut.
2. Replace the three aluminum spacers when a G-type nut is removed.
3. Do not touch any GC parts other than the injection port connections and parts (1) - (7) of the flow path diagram of Section 10.6.1 "GC Carrier Gas Leak Check", page 228. Contact your Shimadzu Service Representative for maintenance in other areas.

Procedure for Pressure-Sealing Carrier Gas

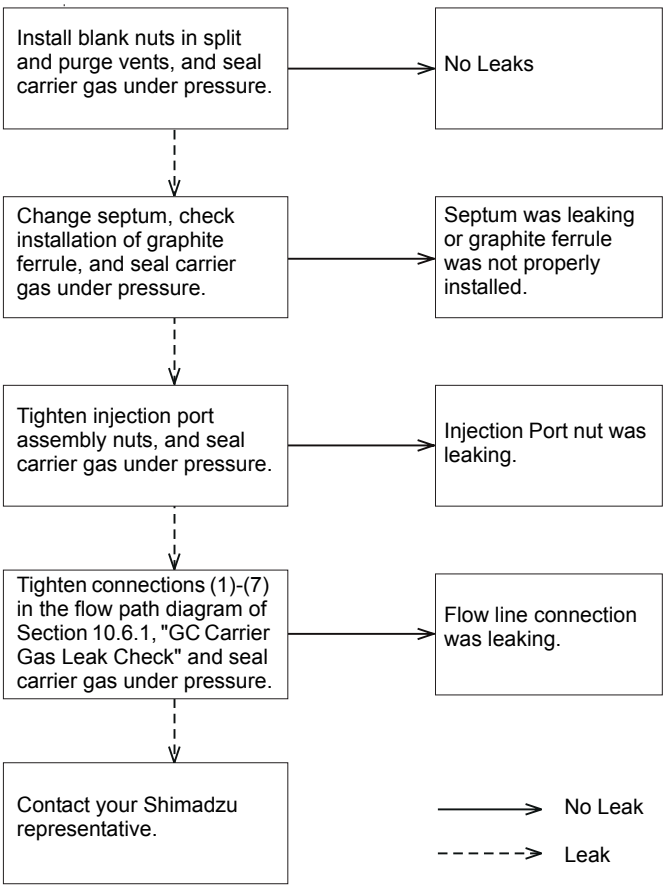


Figure 10.14 Procedure for Pressure-Sealing Carrier Gas

10.6.2 MS Vacuum Leak Check

This section describes MS vacuum leak check procedures. Leaks can be large enough to take the rotary pump off-line, or small enough to allow the turbomolecular pump to function almost normally.

Ensure that the O-ring is properly installed on the MS door. The O-ring seals the connection between two surfaces, preventing vacuum leaks and maintaining the proper vacuum in the MS.

When the MS door is opened to perform maintenance, observe the following precautions to avoid leaks.

Note

Observe the following precautions to prevent vacuum leaks:

- Do not forget to install the O-ring.
- Install the O-ring properly.
- Remove any foreign material from the O-ring.
- Remove foreign material, including pieces of O-ring, from sealing surfaces.
- Do not scratch sealing surfaces.
- Do not forget to install the Vespel ferrule where the column connects to the interface.
- Firmly tighten the column interface nut.
- Change the GC septum on a regular basis (around 80 to 100 injections).

If the rotary or turbomolecular pumps are not functioning properly:

1. The sound of air exhausting from the rotary pump does not diminish after a few minutes.
2. The turbomolecular pump operates, but the vacuum system power shuts down after about 5 minutes.

If the situation in step 2 occurs, a vacuum leak is indicated. Wait for the MS to vent, then check the front door and the connection between the column and interface according to the above precautions for preventing vacuum leaks. If a vacuum leak occurs in a location other than the front door or connection between the column and interface, contact your Shimadzu Service Representative.

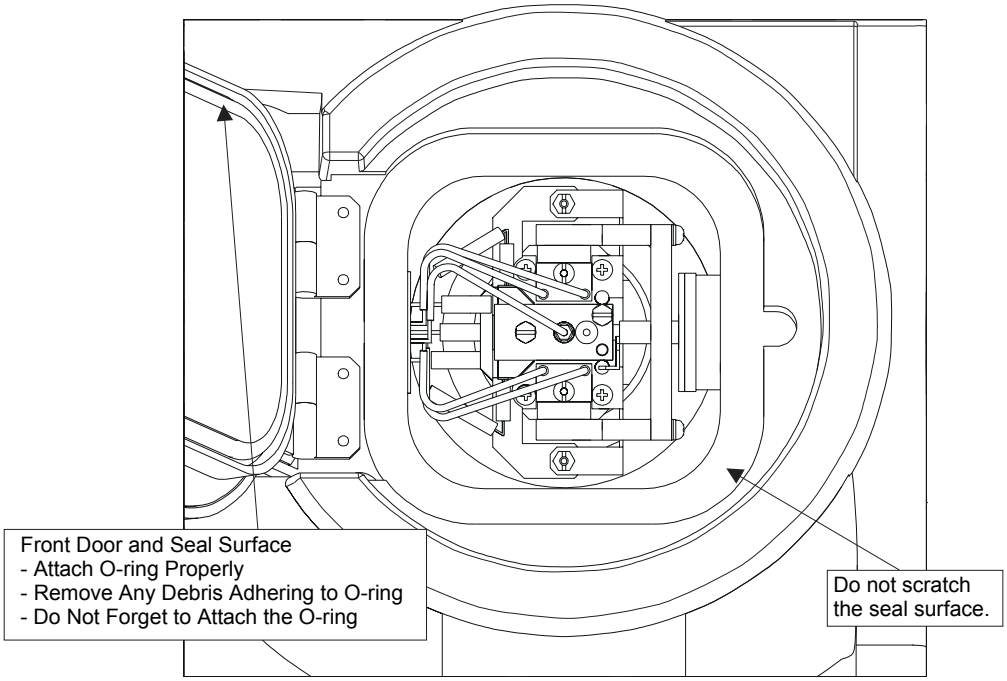


Figure 10.15 Front Door and Sealing Surfaces

10.6.3 Vacuum Leak Check Using Peak Monitor

Vacuum leaks too small to prevent proper operation of the instrument can still affect the instrument performance. This section describes the procedure for checking for those leaks. The operations described in this section can be used provided that the instrument and turbomolecular pump are functioning properly and the Turbo Pump light is green in the "Vacuum Control" dialog box.

This method monitors the water (m/z 18) and nitrogen (m/z 28) peaks from the "Peak Monitor" window. The height of these two peaks is compared to check for small vacuum leaks.

1. Click the GCMS Real Time Analysis Assistant Bar **Tuning** icon. The "Tuning" window opens.

2. Click the Assistant Bar **Peak Monitor View** icon. This opens the "Peak Monitor" window.

3. Set the m/z of the ions to be monitored to 18 (water) and 28 (nitrogen), or choose the Water, Air Monitor Group.
4. Set the detector voltage to about 1.00 - 1.50 kV so that the peaks for m/z 18 and m/z 28 are visible.
5. Turn on the filament.
6. Compare the peak heights for m/z 18 and m/z 28. If the height of m/z 28 is more than twice that of m/z 18, a vacuum leak is indicated.
7. When a vacuum leak is detected, shut down the instrument according to Section 2.4.2 "Shutting Down the Instrument", page 26. Turn off the main instrument switch.
8. Follow the precautions for vacuum leak prevention described in Section 10.6.2 "MS Vacuum Leak Check", page 232.

Note

If an optional accessory for sample introduction is connected, the vacuum leak check may not meet standards, even if there is not leakage in the GC/MS. Insufficient purging may result in a high 28 m/z to 18 m/z ratio. When this occurs, increase the total flow of the GC to around 200 mL/min to purge.

10.6.4 Vacuum Check using Petroleum Ether

Note

Precautions when Performing Vacuum Leak Check with Petroleum Ether

1. Use only petroleum ether.
2. Do not spill petroleum ether on any electronics.
3. Do not allow petroleum ether to contact any site other than contact surface between the interface and column.

This procedure is based on the suction produced when a vacuum leak occurs in the MS. If there is a leak, the petroleum ether applied to the contact surface between the column and the interface will be detected at that time in the "Peak Monitor" window, by the m/z 43 peak. The vacuum check is performed by monitoring the fluctuations of this m/z 43 peak.

1. Click on the **Tuning** icon in the GCMS analysis assistant bar. The "Tuning" window opens.

2. Click on the **Peak Monitor View** icon in the Assistant bar to open the "Peak Monitor" window.

3. Set the m/z for the ion to be monitored in the "Peak Monitor" window to 43 (fragment ion of petroleum ether). Set the magnification factor to 50 - 100.
4. Set the detector voltage to 1.00 - 1.50 kV.
5. Turn on the filament.
6. Fill a syringe with petroleum ether.
7. Apply petroleum ether to the contact surfaces of the front door and between the column and interface.
8. Observe the m/z 43 peak in the "Peak Monitor" window. If the m/z 43 peak fluctuates greatly (increases), there is vacuum leak at the petroleum ether application site.
9. Repeat the procedures described in steps 7 and 8. If there is no change in the m/z 43 peak, there is no leak.
10. If a vacuum leak has been detected, follow the precautions for vacuum leak prevention described in Section 10.6.2 "MS Vacuum Leak Check", page 232.

10.7 Software Installation

This section describes reinstallation of GCMSsolution when this or other applications are not functioning properly.

10.7.1 Before Installation

Check the following items before installing the software.

Installation Disks
The installation program is provided on CD-ROM.

This program decompresses program files stored on the CD-ROM and copies them to the PC hard drive during installation.

> **Note**
>
> The software cannot be installed by copying the contents of the CD-ROM to the hard disk of the PC. Installation must be performed by the method described here.

Windows Installation
Windows 2000, Windows XP or Windows Vista is required.

On a new PC, check to make sure that Windows has been installed on the PC hard drive and is functioning properly.

Internet Explorer version 3.02 or higher is required.

> **Note**
>
> If the GCMSsolution software is being reinstalled, first uninstall the GCMSsolution software. Refer to Section 10.7.3 "GCMSsolution Uninstall", page 242.

10.7.2 GCMSsolution Installation

1. Start the PC and log in to Windows. Place the GCMSsolution installation disk in the CD-ROM drive.

   If the "Found New Hardware Wizard" window is displayed after starting Windows, click the **Cancel** button and close the window. This means that the driver for MS is not installed in the PC. Install the driver after the GCMSsolution Installation. Refer to Section 10.8.4 "Installation of the Driver for MS (Windows 2000)", page 254.

   GCMSsolution Setup launches automatically, and the "Welcome" window is displayed.

   For Windows Vista, the dialog box "A program needs your permission to continue" may be displayed. In this case, click the **Continue** button to proceed.

Note

If the "Welcome" window does not open automatically, select Run from the Windows Start menu. For Windows Vista, select the **Start menu > All Programs > Accessories > Run**. Enter E:\ GCMSsolution\Setup.exe as the name of the program to execute (where E: is the CD-ROM drive), and click the **OK** button.

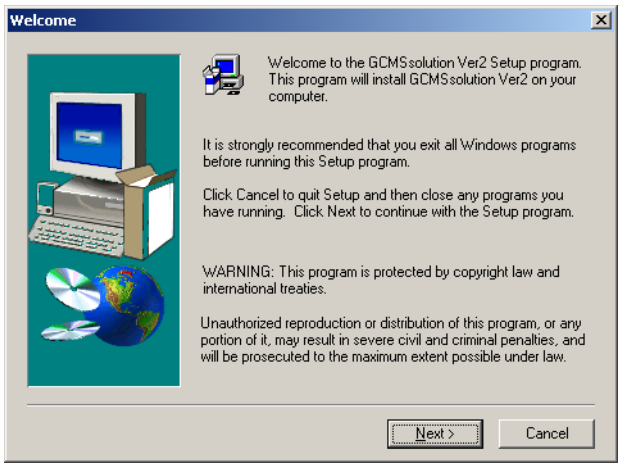


Figure 10.16 "Welcome" Window

2. Read the contents of the "Welcome" window, then click the **Next** button.

Note

When the **OK** button in the "Welcome" window is clicked, and the Fail to detect DAO message is displayed, click the **OK** button to stop the installation. Refer to Section 10.7.4 "DAO Installation", page 245, and after installation of "Data Access Objects (DAO) Setup", try installing GCMSsolution again.

The "Detecting Internet Explorer" window is displayed.

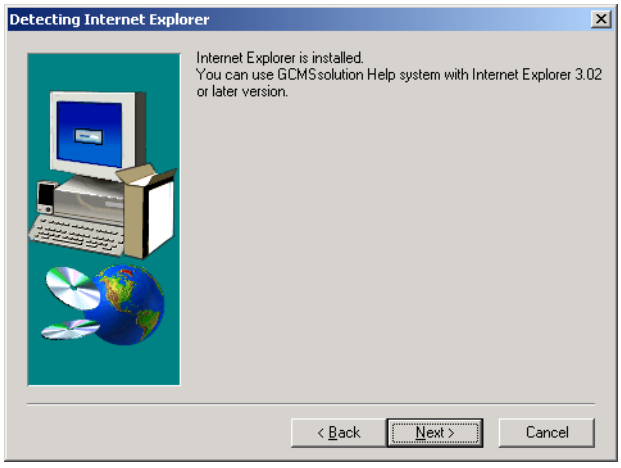


Figure 10.17 "Detecting Internet Explorer" Window

3. Click the **OK** button in the "Detecting Internet Explorer" window.
The screen switches to the "Installing HTML Help" window.

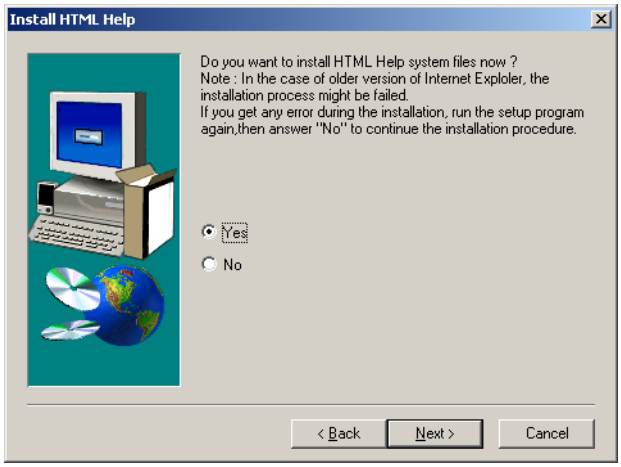


Figure 10.18 "Installing HTML Help" Window

4. Select **Yes** and click the **Next** button.
The "User Authentication Mode Select" window is displayed.

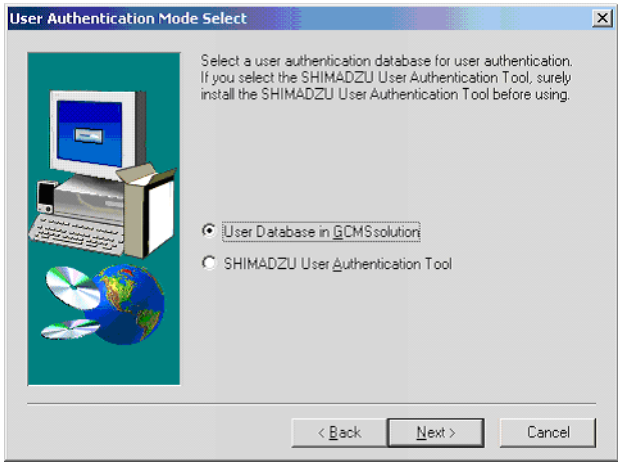


Figure 10.19 "User Authentication Mode Select" Window

5. Specify the user authentication mode. When not using the CLASS-Agent, select "User Database in GCMSsolution". When using the CLASS-Agent, select an arbitrary user authentication mode by referring to Section "2.9.4, 3. Using Shimadzu User Authentication Tool".
The "Do you want to use the GC detector?" window is displayed.

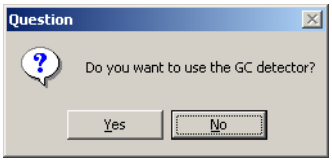


Figure 10.20 "Do you want to use the GC detector?" Window

6. The installation of GCsolution starts after clicking the **Yes** button. If the No button is clicked or the installation finishes, the screen returns to GCMSsolution Installation.
The "User Information" window of GCMSsolution is displayed.

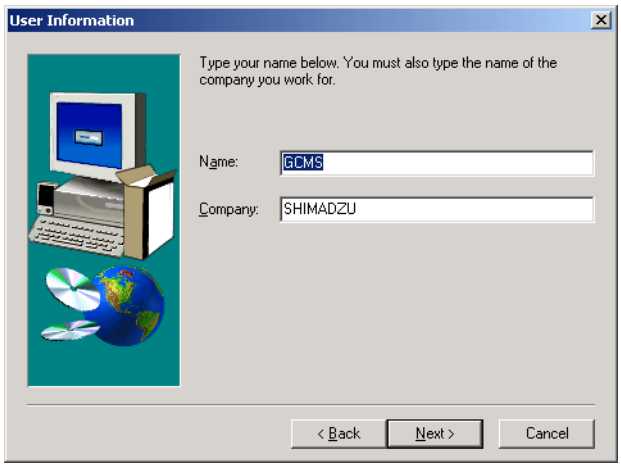


Figure 10.21 "User Information" Window

7. Enter the User Name and Company in the "User Information" window. Then click the **Next** button.
The "Choose Destination Location" window is displayed.

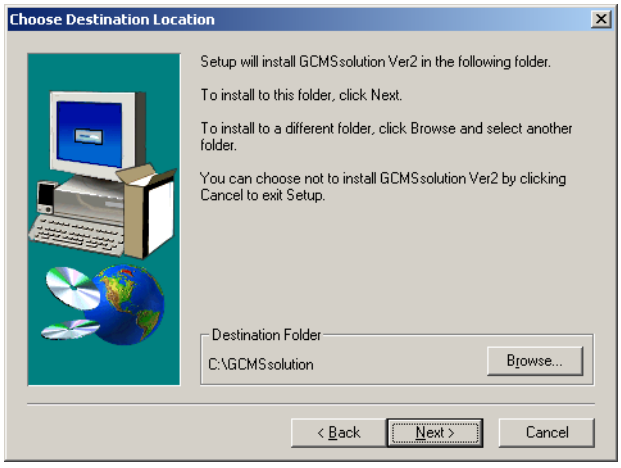


Figure 10.22 "Choose Destination Location" Window

8. The GCMSsolution application will be installed in the Destination Folder indicated in the "Choose Destination Location" window. The default directory is commonly selected.

Note

To change the destination folder, click the **Browse** button of the "Choose Destination Location" window.
The "Choose Folder" dialog box opens.

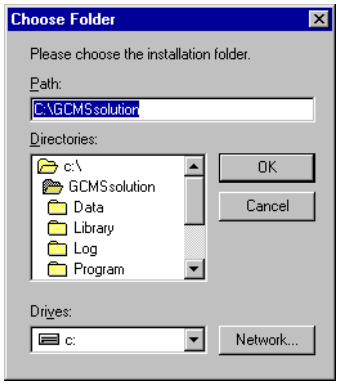


Figure 10.23 "Choose Folder" Dialog Box

Enter the full path in the Path text box, or double click the Folder list box to select the installation folder.

To change the drive, click the Drive box and select the drive.

Note

This note is applied in either of the following cases:

- While using Windows XP Professional as the OS, multiple users, including limited users (members of the Users group) or members of the Power Users group, share the GCMSsolution.
- While using Windows Vista as the OS, multiple users share the GCMSsolution.

In the conditions above, measurement data or method files created by a Windows user may be unavailable to other Windows users. This is because (1) default security settings for newly created folders are more strict in Windows XP, and (2) the user account control (UAC) functions introduced in Windows Vista effects the relevant operation. To avoid the problem, the GCMSsolution automatically changes the security settings for the installation folder and its subfolders during installation, so that all Windows users can share the data files, method files and so on.

Therefore, if you newly create a data folder in any place other than the Data folder (or its subfolders) designated during installation, you may become unable to create subfolders or save files in that data folder, or files may become unavailable to other users. In such cases, change the appropriate security settings for the folder created, using the Windows system functions.

Click the **Next** button, and the "Copying Program Files" status message is displayed. The program files are installed.

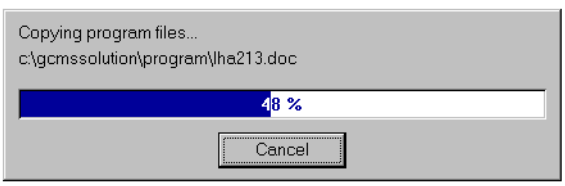


Figure 10.24 "Copying Program Files" Status Message

Note

To stop the installation, click the **Cancel** button.
The "Exit Setup" dialog box is displayed.

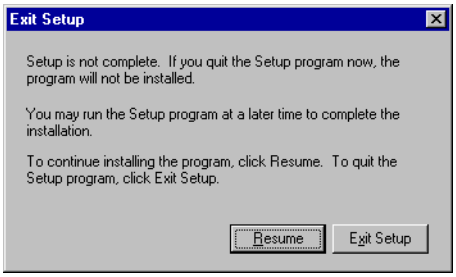


Figure 10.25 "Exit Setup" Dialog Box

To resume installation, click the **Resume** button.

To stop the installation, click the **Exit Setup** button. Installation is stopped, and GCMSsolution Setup exits.

9. When installation is completed, the "Setup Complete" window opens.

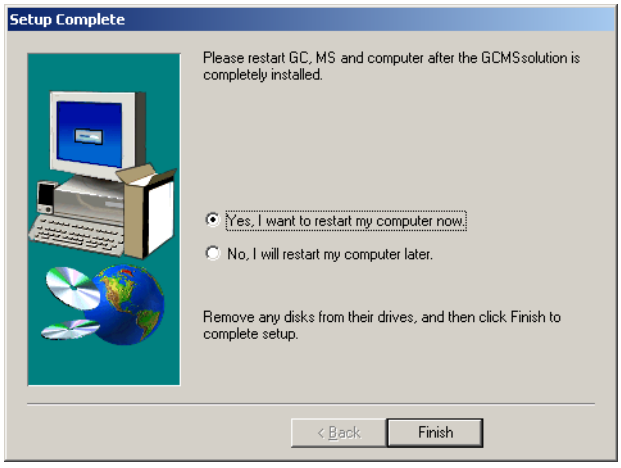


Figure 10.26 "Setup Complete" Window

10. Click the **Finish** button in the "Setup Complete" window. The "Setup Complete" window closes. GCMSsolution setup is completed.

Power to the GC, MS and all other accessories can be turned back on.

10.7.3 GCMSsolution Uninstall

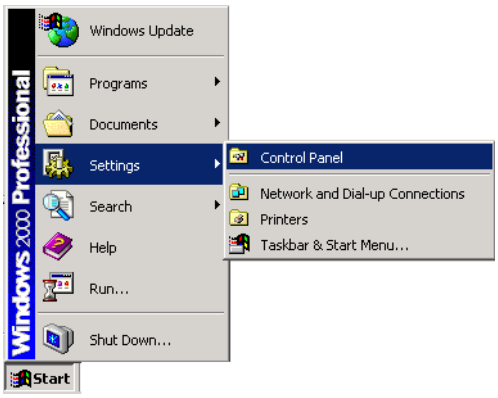


Figure 10.27 Windows Start Menu

1. From the **Windows Start menu**, select **Control Panel**. The "Control Panel" window opens.

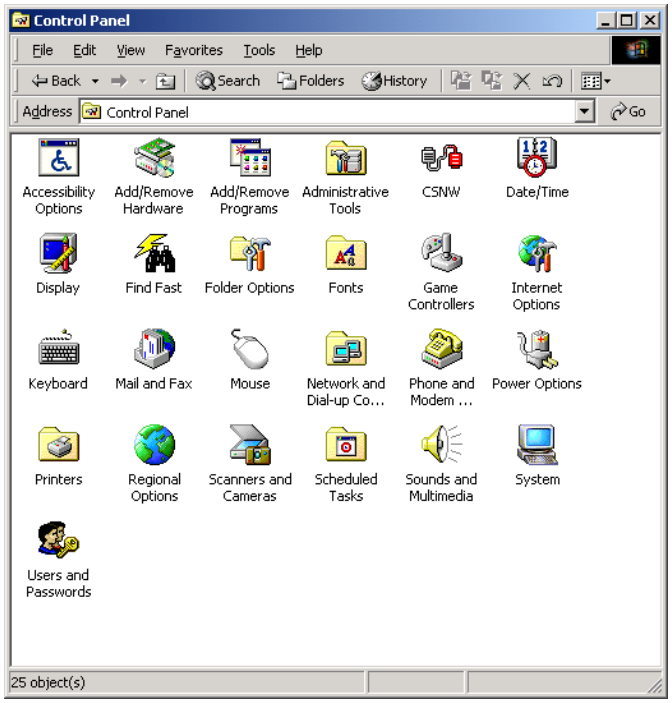


Figure 10.28 Windows Control Panel

2. Double-click the **Add/Remove Programs** icon.

The "Add/Remove Programs" window opens.
For Windows Vista, switch **Control Panel** to Classic View, and double-click **Programs and Features**.

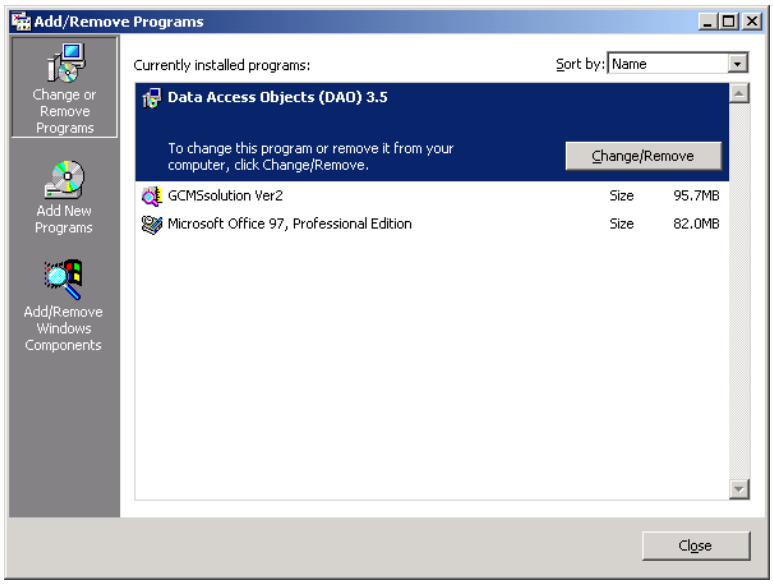


Figure 10.29 "Add/Remove Programs" Window

3. Click to select "GCMSsolution Ver. 2" from the list box at the bottom of the Currently installed programs tab. Click the **Change/Remove** button.

If GCsolution has been installed, uninstall it as well. The procedure is the same as that for GCMSsolution.

4. The "Confirm File Deletion" message is displayed. Confirm that the program should be removed, or cancel by clicking the **No** button.

Figure 10.30 Confirm File Deletion Message

Note

During the uninstall, the "Remove Shared File?" dialog box asks whether to remove shared files. All files indicated here are not used except by GSMSsolution; therefore, click the **Yes To All** button.

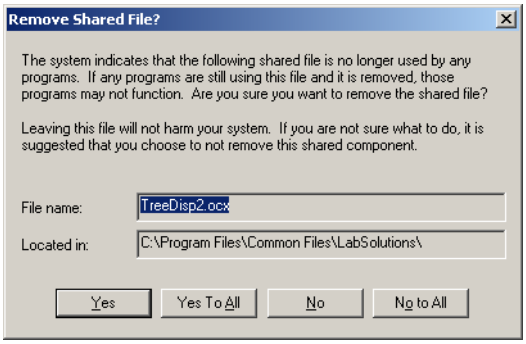


Figure 10.31 "Remove Shared File?" Dialog Box

When the **Yes to All** button is clicked, all shared files not used by the system will be deleted. Click the **Yes** button to delete the currently displayed file.

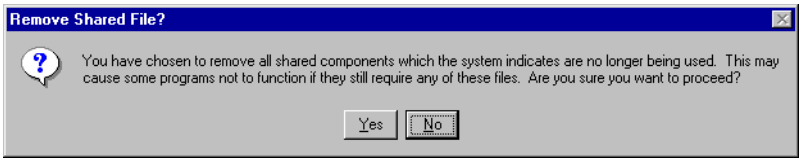


Figure 10.32 "Remove Shared Files?" Confirmation

The "Remove Programs From Your Computer" dialog box is displayed, and GCMSsolution program files are removed.

Figure 10.33 "Remove Programs From Your Computer" Dialog Box

5. When the uninstall procedure is completed, the **OK** button of the Remove Program From Your Computer status box becomes active. Click the **OK** button to complete the GCMSsolution uninstall process.

> **Note**
>
> To reinstall GCMSsolution after un-installing it, first, restart your computer before reinstalling GCMSsolution.

10.7.4 DAO Installation

1. Double-click the **Add/Remove Programs** icon in the "Control Panel" window to open the "Add/Remove Programs" dialog box.
For Windows Vista, click "Uninstall a program".

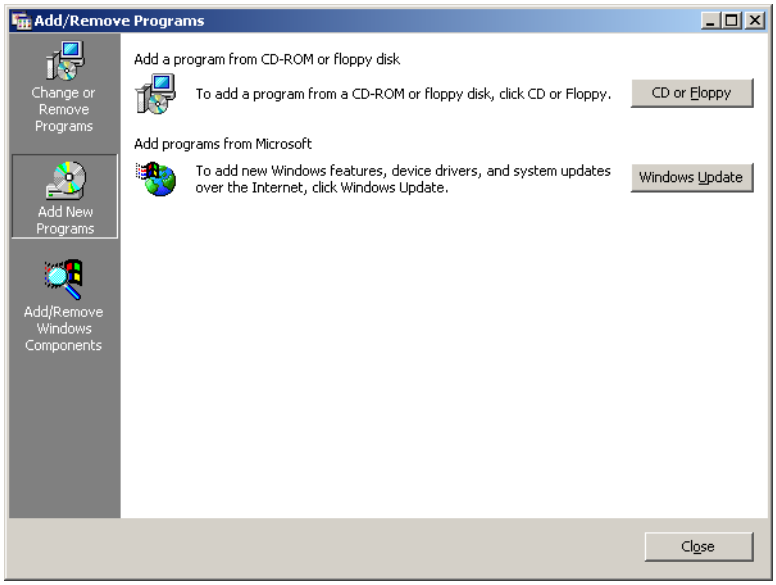


Figure 10.34 "Add/Remove Programs" Window

2. Click the **Add New Programs** button and then click the **CD or Floppy** button.
The "Install Program From Floppy Disk or CD-ROM" window is displayed.

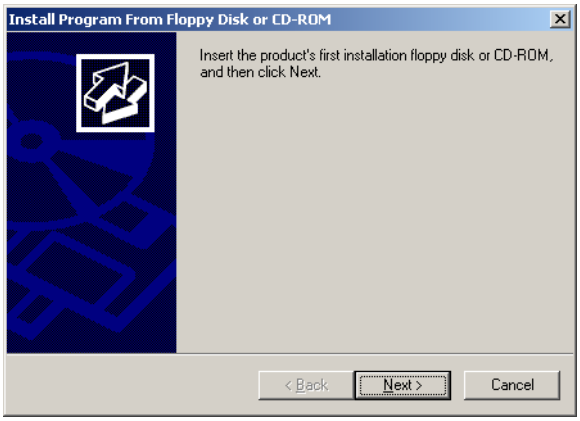


Figure 10.35 "Install Program from Floppy Disk or CD-ROM" Window

3. Click the **Next** button in the "Install Program From Floppy Disk or CD-ROM" window. The "Run Installation Program" window is displayed.

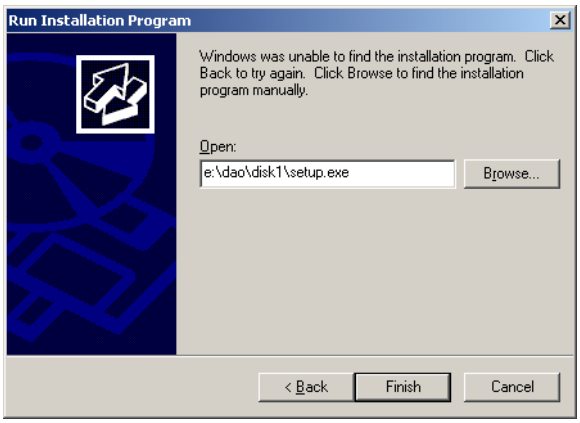


Figure 10.36 "Run Installation Program" Window

4. Place the GCMSsolution installation disk in the CD-ROM drive. If the GCMSsolution installation window is displayed, click the **Cancel** button to exit the installation. Enter the full path of the DAO installation program in the Open text box. For example, if the CD-ROM drive is the E drive, input the following:

   E:\ Dao\Disk1\Setup.exe

   Click the **Finish** button.

   "Data Access Objects (DAO) Setup" launches and the "Welcome" window is displayed.

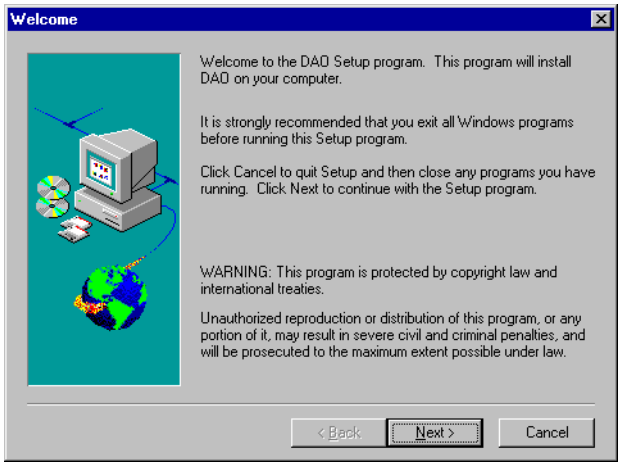


Figure 10.37 DAO Installation "Welcome" Window

5. Read the contents of the "Welcome" window and click the **Next** button.
The "Select Components" window is displayed.

Figure 10.38 "Select Components" Window

6. Select only the Jet 3.5 check box from the "Select Components" window, and click the **Next** button.
The next "Select Components" window opens which allows the selection of the data format to be used for Jet 3.5.

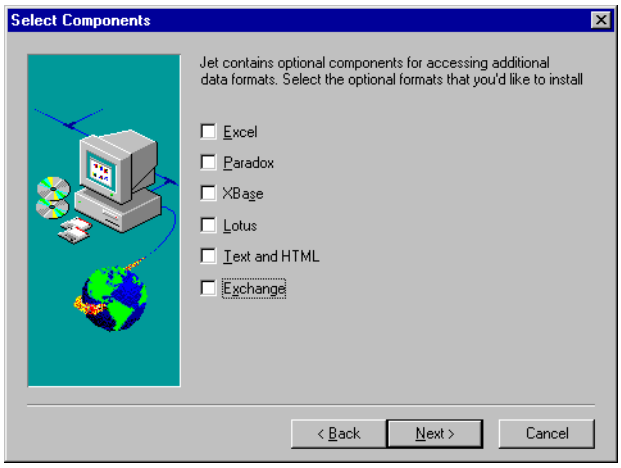


Figure 10.39 The Second "Select Components" Window

7. Deselect all check boxes, and click **Next**.
DAO is installed, and when completed, the Information message is displayed.

Figure 10.40 DAO Installation Completed

8. Click the **OK** button to complete "Data Access Objects (DAO) Setup".

Note

Perform GCMSsolution installation when DAO installation is completed.

Note

The following message can occur during DAO installation, indicating installation failure:
"The OLE automation DLL, OLEAUT32.DLL, could not be found or is an older version that is incompatible with DAO3.5. If you continue DAO will not register properly. Continue anyway?"
Continue with the DAO installation to its completion. Then install GCMSsolution to complete the installation process.
If the GCMSsolution installation fails, reinstall DAO and GCMSsolution.

10.8 PC Interface Board Installation

This section describes the installation of the PC Interface board that connects the MS and PC and the drivers for the PC Interface board and the MS.

10.8.1 Installing the PC Interface Board

Install the PC Interface board in the PC as follows:

1. Turn off power to the PC and peripheral devices (printer, etc.).
2. Disconnect the power cable from the PC.
3. Remove the PC cover. Refer to the manual for the PC.
4. Remove the cover from the PCI expansion slot where the PC Interface board is to be installed. Use a screwdriver to remove the expansion slot screw.
5. Insert the PC Interface board into the PCI expansion slot, and tighten the screw to secure the board.
6. Replace the PC cover.
7. Reconnect the PC power cable.

Note

For a detailed description of this procedure, refer to the user manual included with the PC.

10.8.2 PC Interface Board Driver Installation (Windows 2000)

Driver installation is performed after the interface board is installed in the PC.

1. Turn on the PC. The installation of the driver will start automatically during the operating system boot process.
2. Select **Settings > Control Panel** from the **Start menu**. The "Control Panel" window opens.

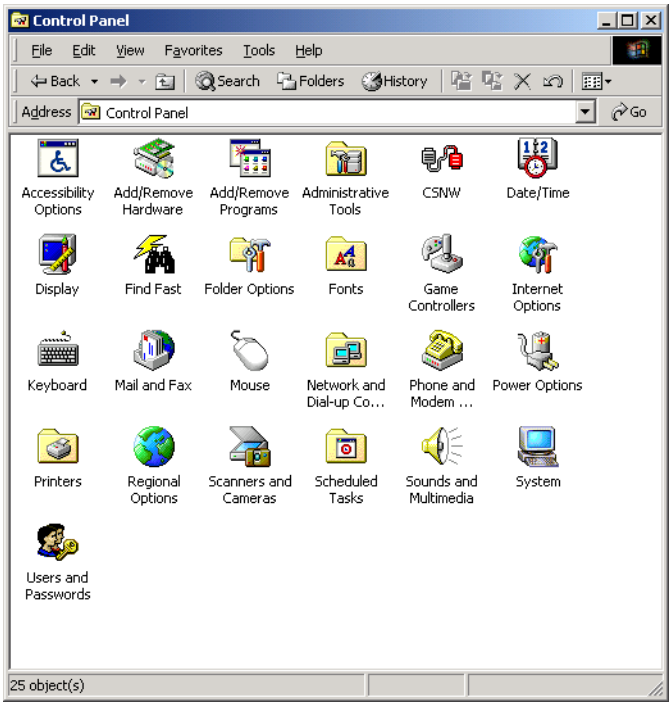


Figure 10.41 "Control Panel" Window

3. Double-click the **System** icon in Control Panel to open the "System Properties" window.

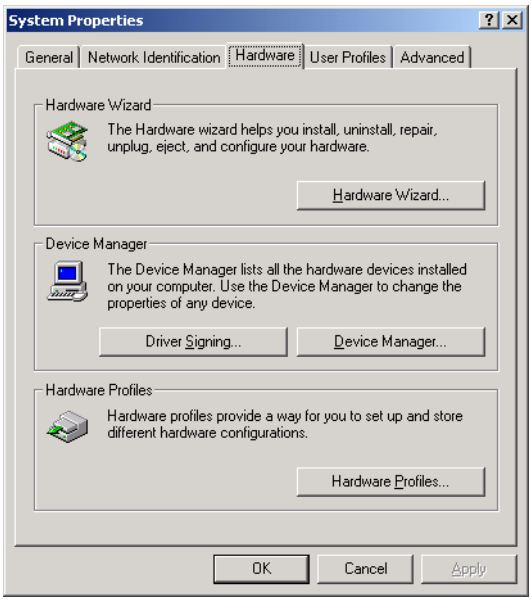


Figure 10.42 "System Properties" Window

Display the Hardware tab and click the **Device Manager** button.

4. The "Device Manager" window opens. Verify that the driver is indicated under "IEEE 1394 Bus host controllers."

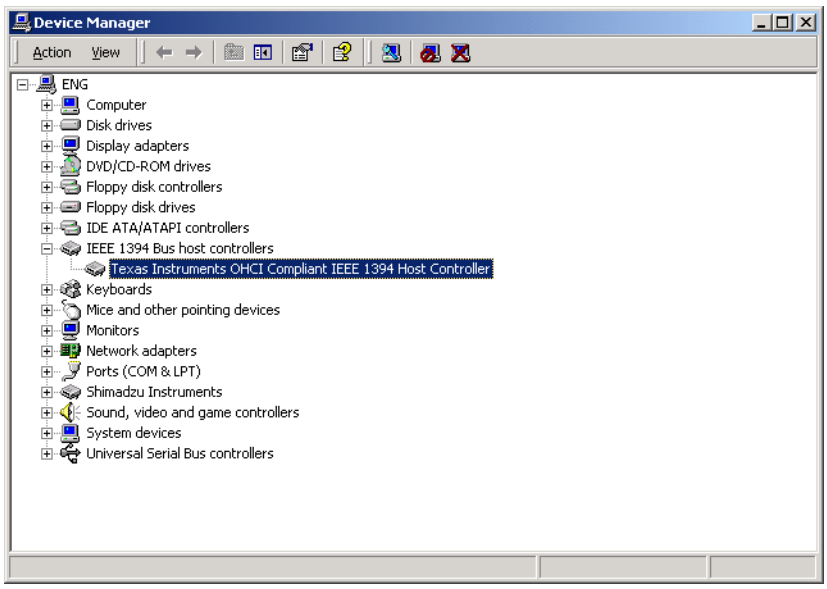


Figure 10.43 "Device Manager" Window

10.8.3 PC Interface Board Driver Installation (Windows XP, Vista)

Driver installation is performed after the interface board is installed in the PC.

1. Turn on the PC. The installation of the driver will start automatically during the operating system boot process.
2. Select **Settings > Control Panel** from the **Start** menu.

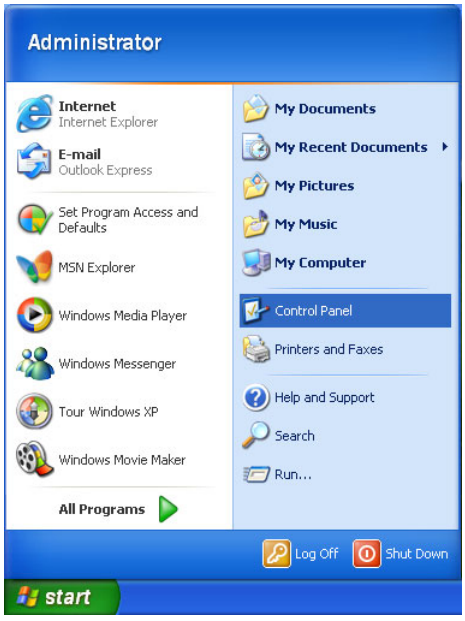


Figure 10.44 "Start" Menu

3. The "Control Panel" window opens. Click the **Performance and Maintenance** icon in Control Panel.
   For Windows Vista, switch to **Classic View**, and click the **Device Manager** icon. Then proceed to step 6.

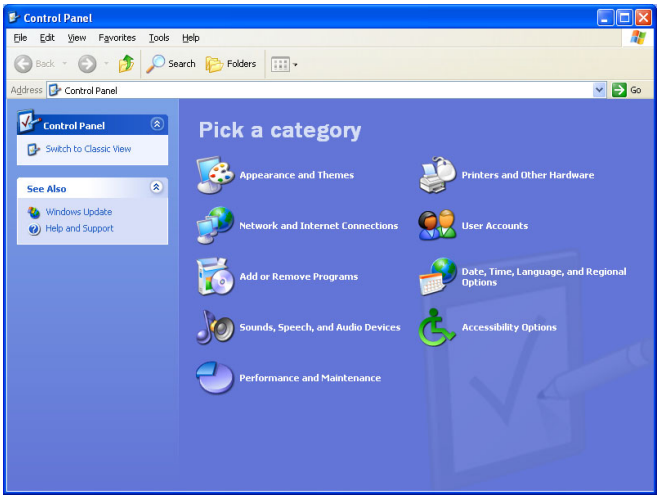


Figure 10.45 "Control Panel" Window

4. Click the **System** icon in the "Performance and Maintenance" window to open the "System Properties" window.

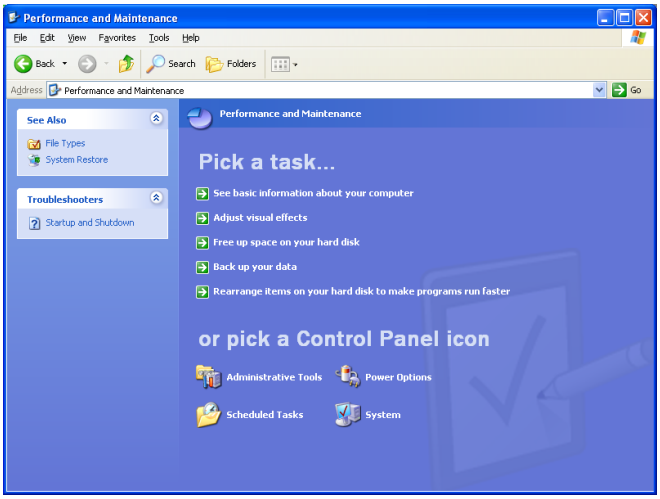


Figure 10.46 "Performance and Maintenance" Window

5. Display the Hardware tab and click the **Device Manager** button.

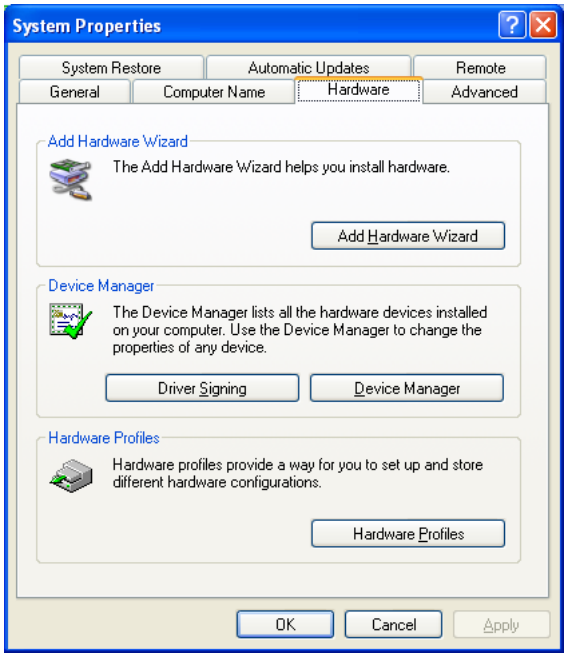


Figure 10.47 "System Properties" Window

6. The "Device Manager" window opens. Verify that the driver is indicated under "IEEE 1394 Bus host controllers".

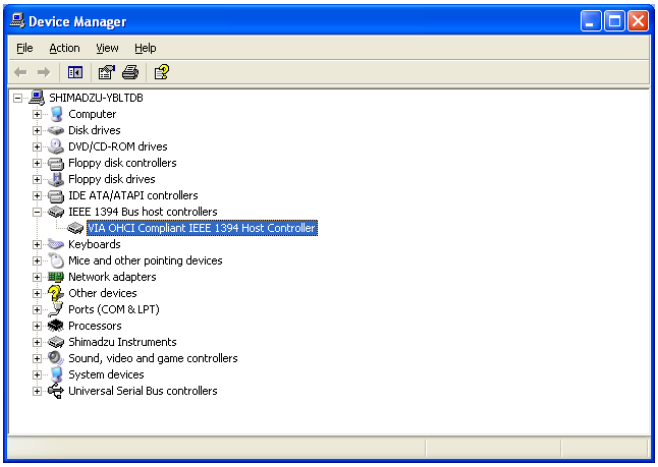


Figure 10.48 "Device Manager" Window

10.8.4 Installation of the Driver for MS (Windows 2000)

1. Connect the MS to the PC Interface board while Windows is running. The "Found New Hardware Wizard" opens automatically.

Figure 10.49 "Found New Hardware" Wizard

2. Click the **Next** button. The screen changes to the next screen of the "Found New Hardware" Wizard - "Install Hardware Device Drivers."

Figure 10.50 "Install Hardware Device Drivers" Screen

Select "Display a list of the known drivers for this device so that I can choose a specific driver" and click the **Next** button.

3. The screen changes to the next screen of the "Found New Hardware" Wizard - the "Hardware Type" screen.

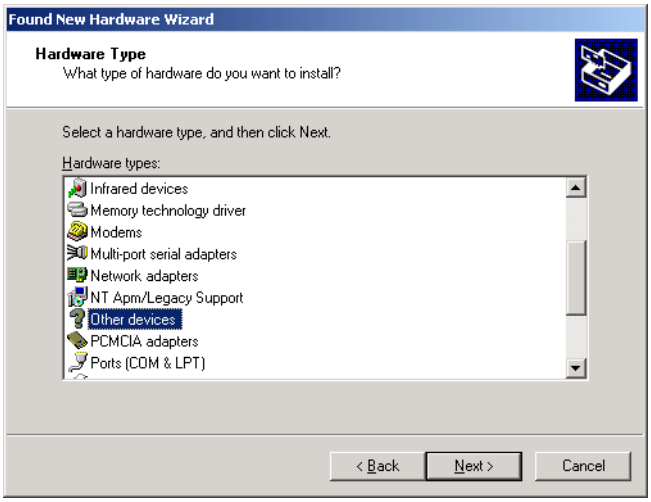


Figure 10.51 "Hardware Type" Screen

Select "Other Devices" and click the **Next** button.

4. The screen changes to the next screen of the "Found New Hardware" Wizard - the "Select a Device Driver" screen.

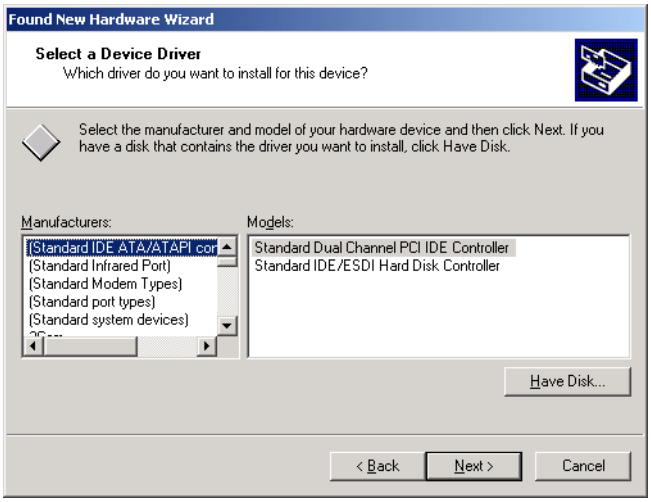


Figure 10.52 "Select a Device Driver" Screen

Click the **Have Disk** button.

5. The "Install From Disk" window opens.
Place the GCMSsolution installation disk in the CD-ROM drive. If the GCMSsolution installation window is displayed, click the Cancel button to exit installation.

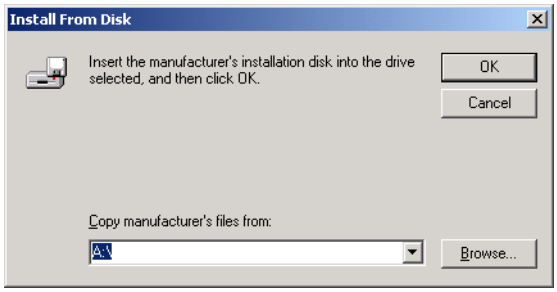


Figure 10.53 "Install From Disk" Window

Type E:\Driver (E: is CD-ROM Drive) in "Copy manufacturer's files from" text box and click the **OK** button.

6. The screen returns to the "Select a Device Driver" screen.

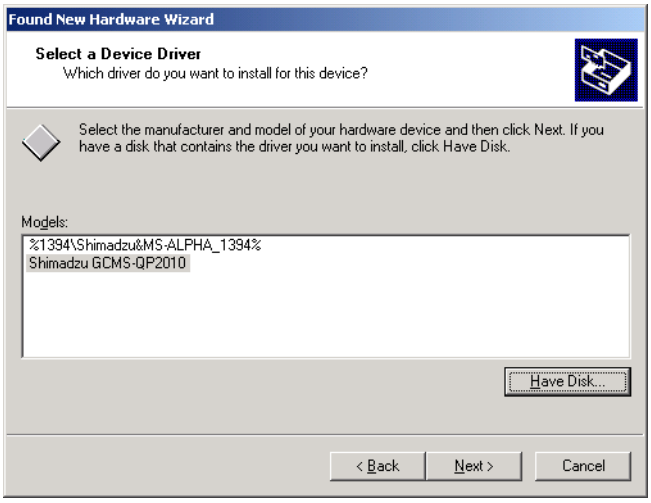


Figure 10.54 "Select a Device Driver" Screen

Select "Shimadzu GCMS-QP2010" and click the **Next** button. If the "Update Driver Warning" window opens, click the **Yes** button.

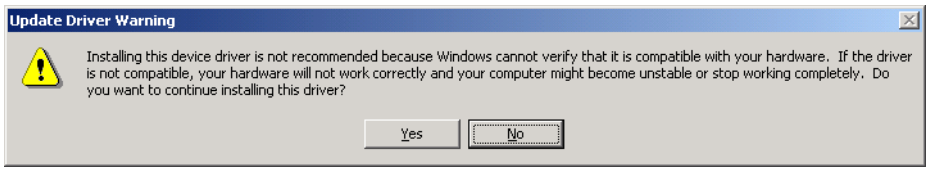


Figure 10.55 "Update Driver Warning" Window

7. The screen changes to the next screen of the "Found New Hardware" Wizard - the "Start Device Driver Installation" screen.

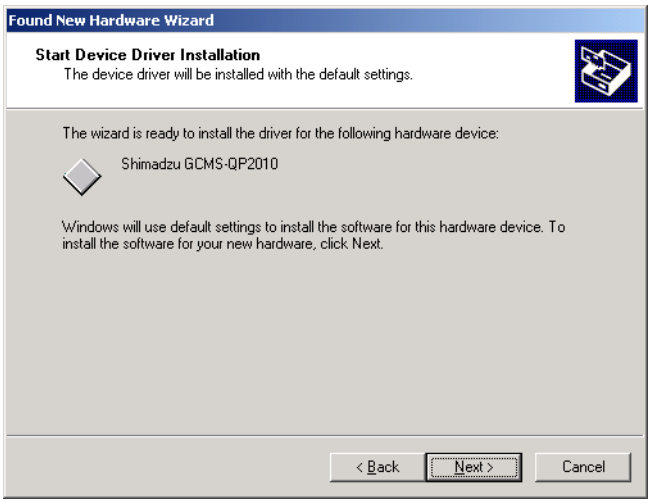


Figure 10.56 "Start Device Driver Installation" Screen

Click the **Next** button.

8. The screen changes to the last screen of the "Found New Hardware" Wizard. Click the **Finish** button.

Figure 10.57 "Completing the Found New Hardware Wizard"

9. Open the "Device Manager" window and verify that "Shimadzu GCMS-QP2010" is indicated under Shimadzu Instruments.
To display the "Device Manager" window, refer to Section 10.8.2 "PC Interface Board Driver Installation (Windows 2000)", page 250.

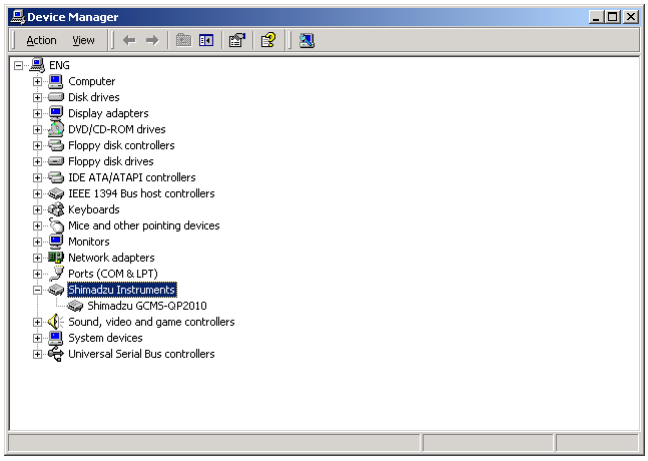


Figure 10.58 "Device Manager" Window

10.8.5 Installation of the Driver for MS (Windows XP)

1. Connect the MS to the PC Interface board while Windows is running. The "Found New Hardware Wizard" opens automatically.

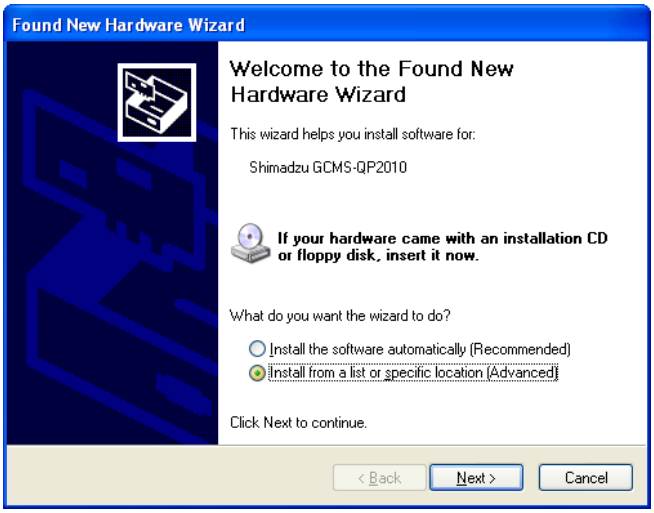


Figure 10.59 "Found New Hardware" Wizard

Select "Install from a list or specific location (Advanced)" and click the **Next** button.

2. The "Please choose your search and installation options" window opens.

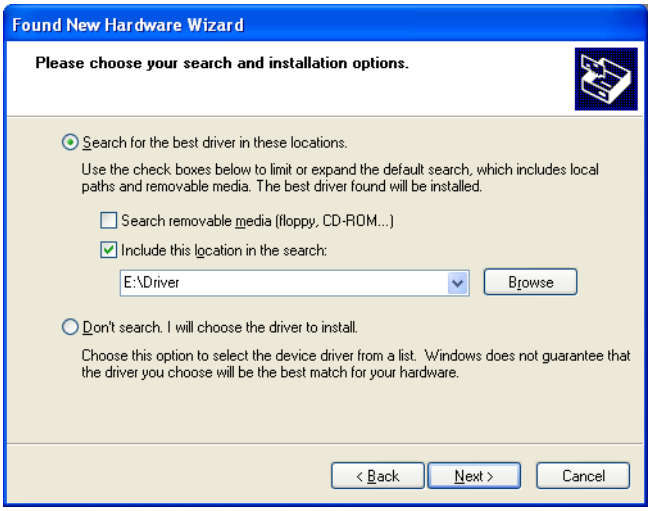


Figure 10.60 "Please choose your search and installation options"

Set the GCMSsolution install disk in the CD-ROM drive. If the GCMSsolution installation window is displayed, click the Cancel button to exit installation.

Check to "Include this location in the search:" check box, type E:\Driver (E: is CD-ROM Drive) in text box.

Click the **Next** button.

3. Installation of device driver starts automatically.

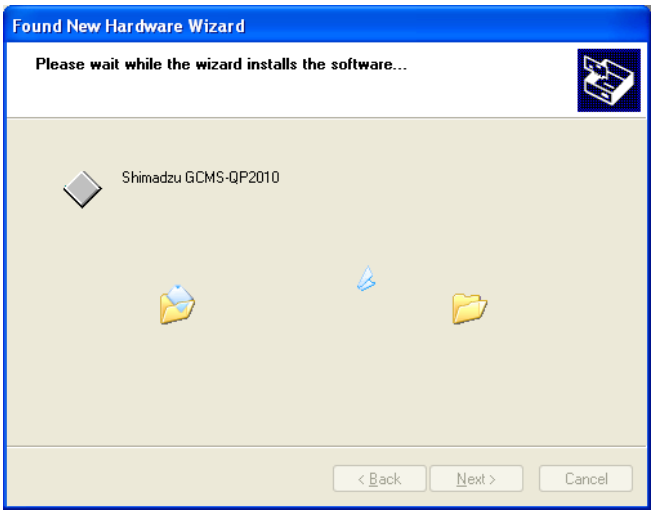


Figure 10.61 "Please wait while the wizard installs the software"

4. When the window below is displayed, click the **Finish** button.

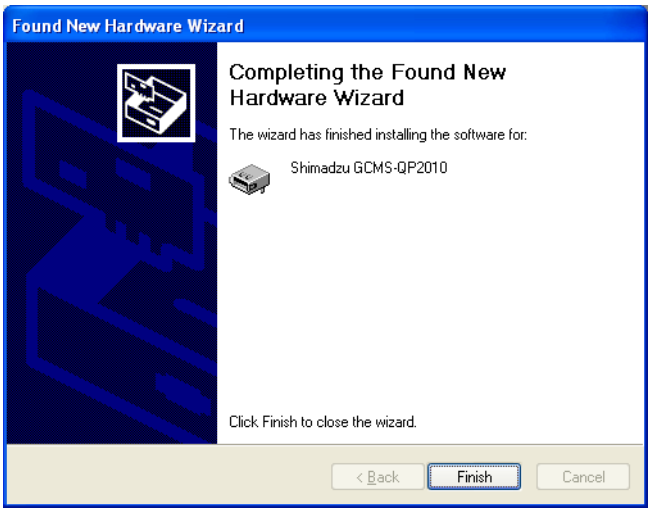


Figure 10.62 "Completing the Found New Hardware Wizard"

5. Open the "Device Manager" window and verify that the "Shimadzu GCMS-QP2010" is indicated under the Shimadzu Instruments.

   To display the "Device Manager" window, refer to Section "10.8.3 PC Interface Board Driver Installation (Windows XP, Vista)".

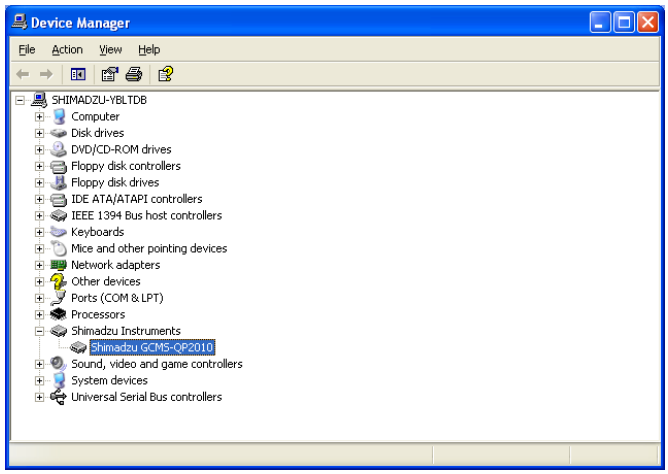


Figure 10.63 "Device Manager" Window

10.8.6 Installation of the Driver for MS (Windows Vista)

Make sure that the GCMSsolution has already been installed onto your computer before installing the driver software for the mass spectrometer.

1. While Windows is running, connect the MS to the PC I/F board. The "Found New Hardware" window is automatically displayed.

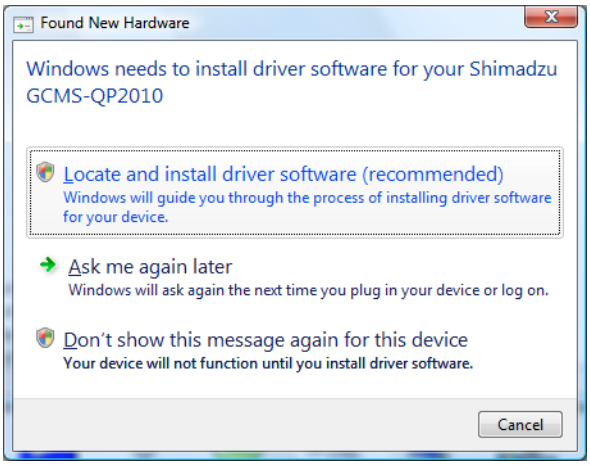


Figure 10.64 "Found New Hardware" Window

Click "Locate and install driver software (recommended)".
In the "User Account Control" window, click the **Continue** button.

2. If a window is displayed asking: "Allow Windows to search online for driver software for your Shimadzu GCMS-QP2010?", click "Don't search online". If this window is not displayed, proceed to step 3.

3. The "Insert the disc that came with your Shimadzu GCMS-QP2010" window will be displayed.
Click "I don't have the disc. Show me other options".

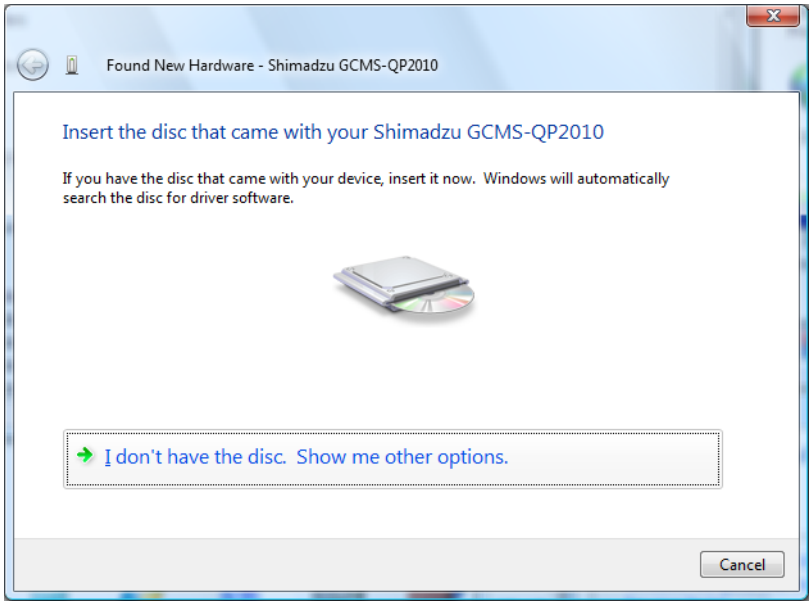


Figure 10.65 "Insert the disc that came with your Shimadzu GCMS-QP2010"

4. The "Windows couldn't find driver software for your device" window will be displayed.
Click "Browse my computer for driver software (advanced)".

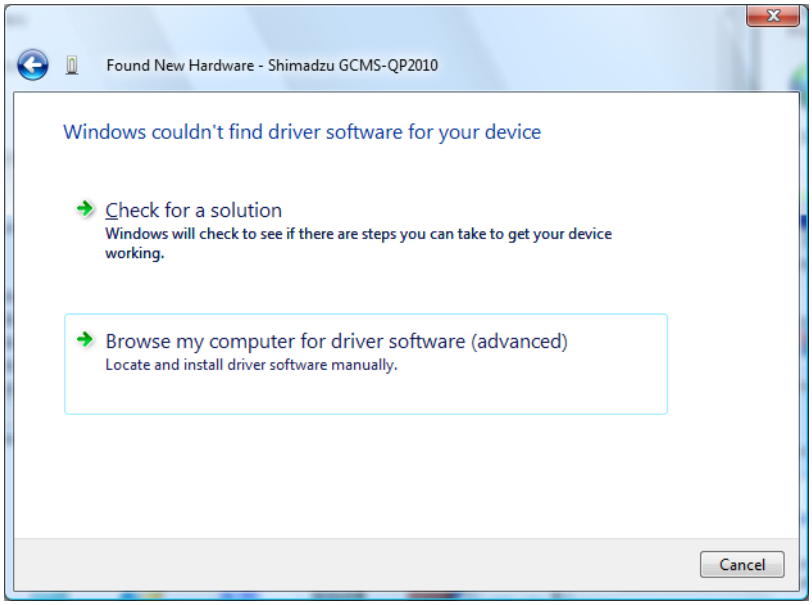


Figure 10.66 "Windows couldn't find driver software for your device"

5. The "Browse for driver software on your computer" is displayed.
Click the **Browse** button and specify "C:\GCMSsolution\Program (C: is the hard drive where the GCMSsolution is installed.)". Click the **Next** button.

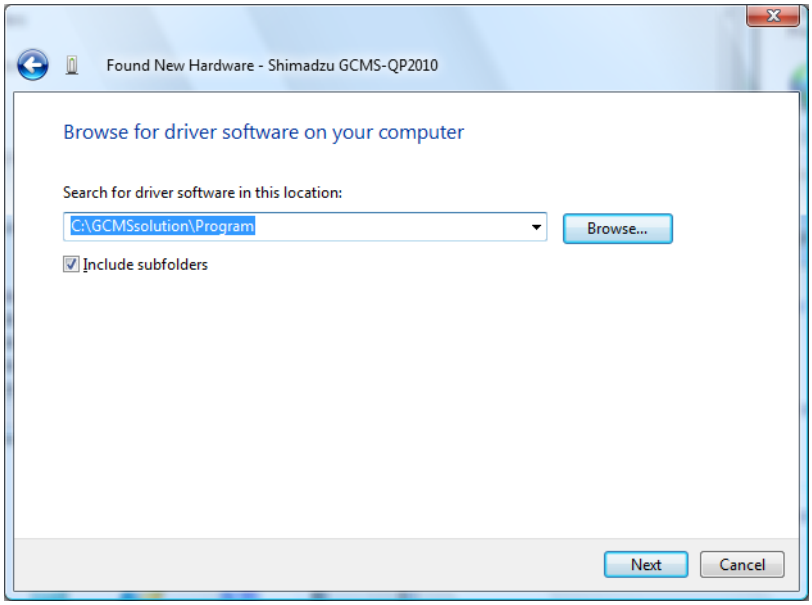


Figure 10.67 "Browse for driver software on your computer"

6. The installation of the device driver begins.

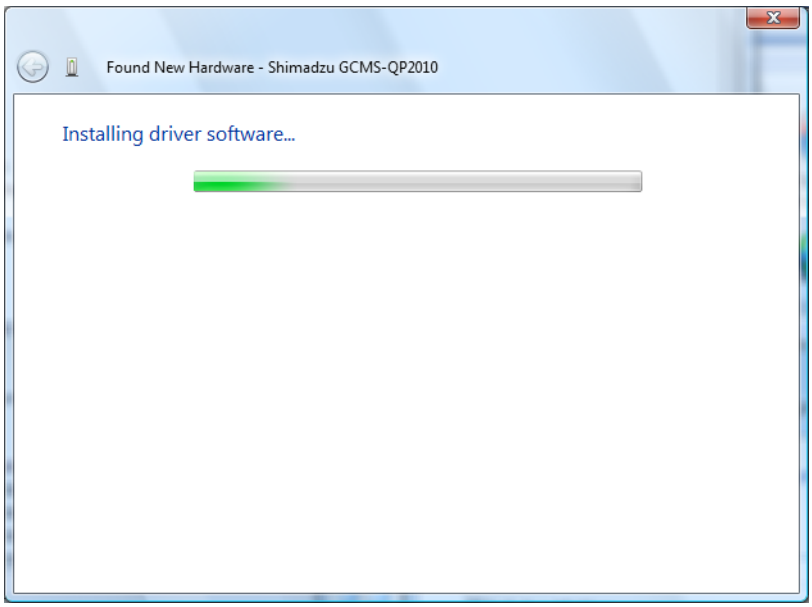


Figure 10.68 "Installing driver software"

If the dialogue box "Would you like to install this device software?" is displayed during installation process, click the **Install** button.

7. The "The software for this device has been successfully installed" window will be displayed. Click the **Close** button.

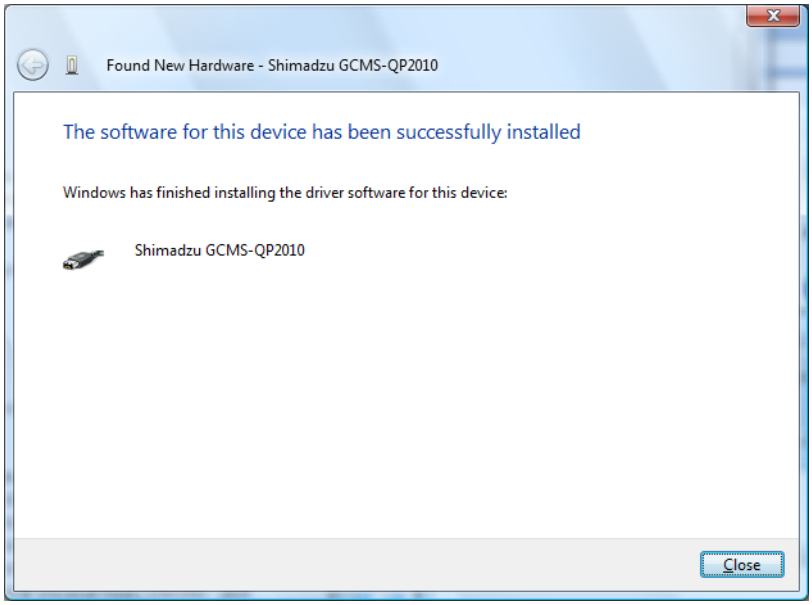


Figure 10.69 "The software for this device has been successfully installed"

11 Troubleshooting

11.1 Operational Problems and Remedial Measures

This section describes operational problems that can occur during an analysis, the causes of these problems and recommended solutions. Contact your Shimadzu Service Representative if symptoms persists after remedial action, or if other problems arise.

11.1.1 Error status indicated by LED on MS

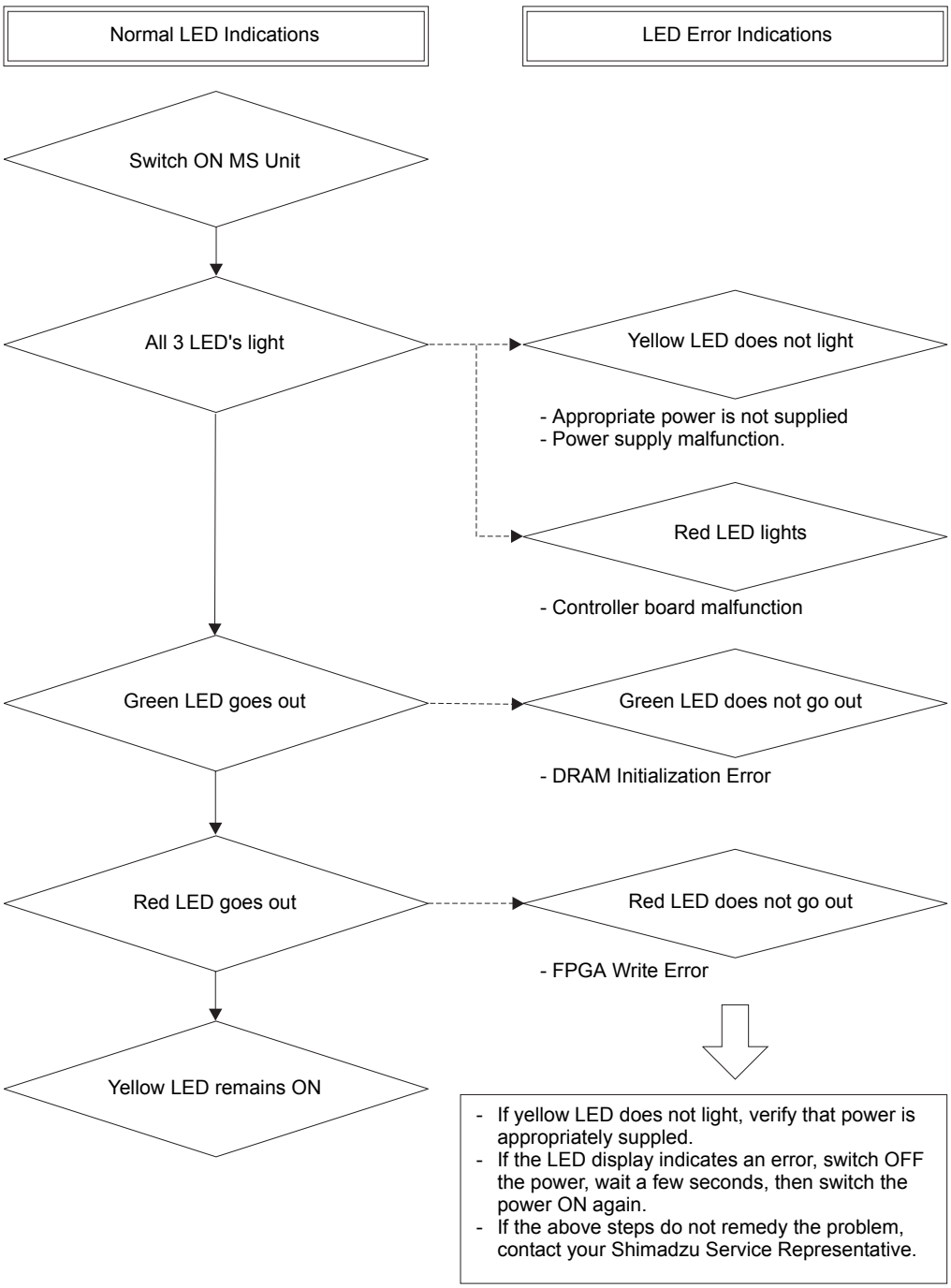


Figure 11.1 LED Error Indications

11.1.2 GCMSsolution Software Startup Errors

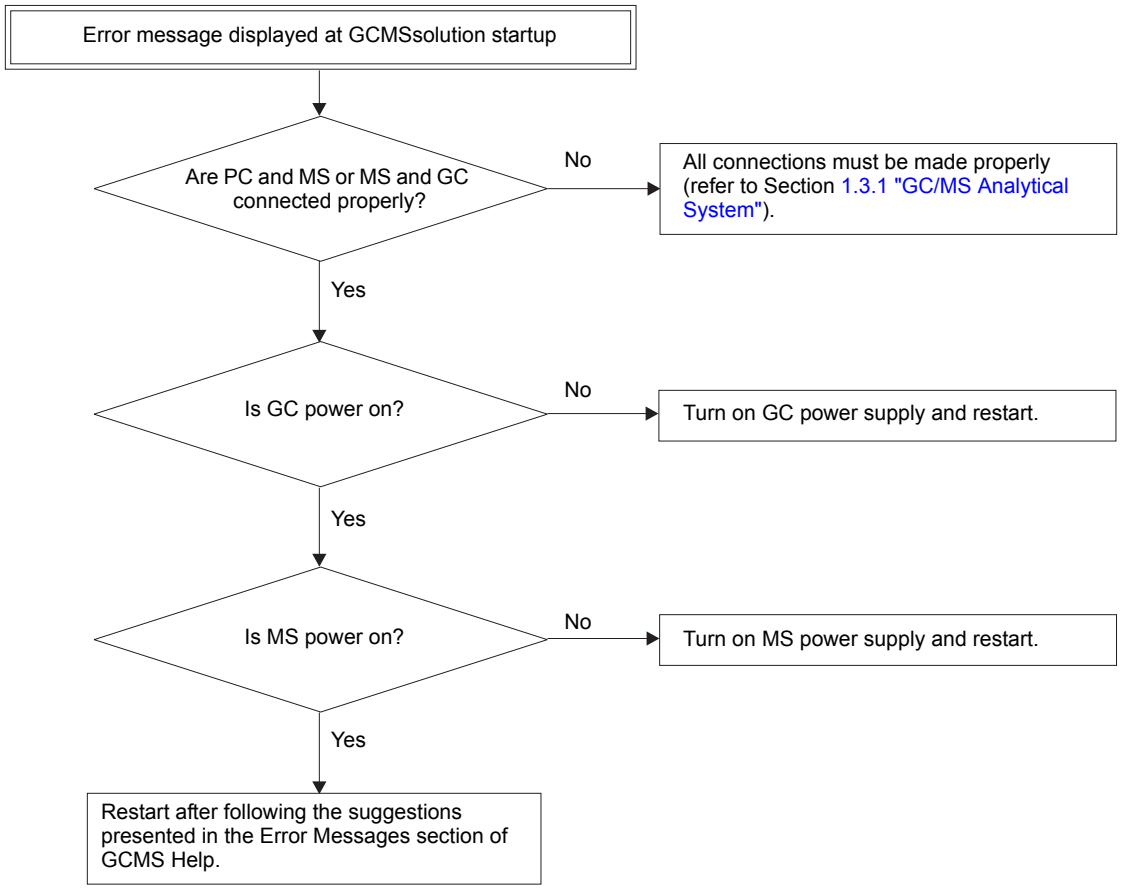


Figure 11.2 Software Startup Errors

11.1.3 Vacuum System Auto Startup Errors

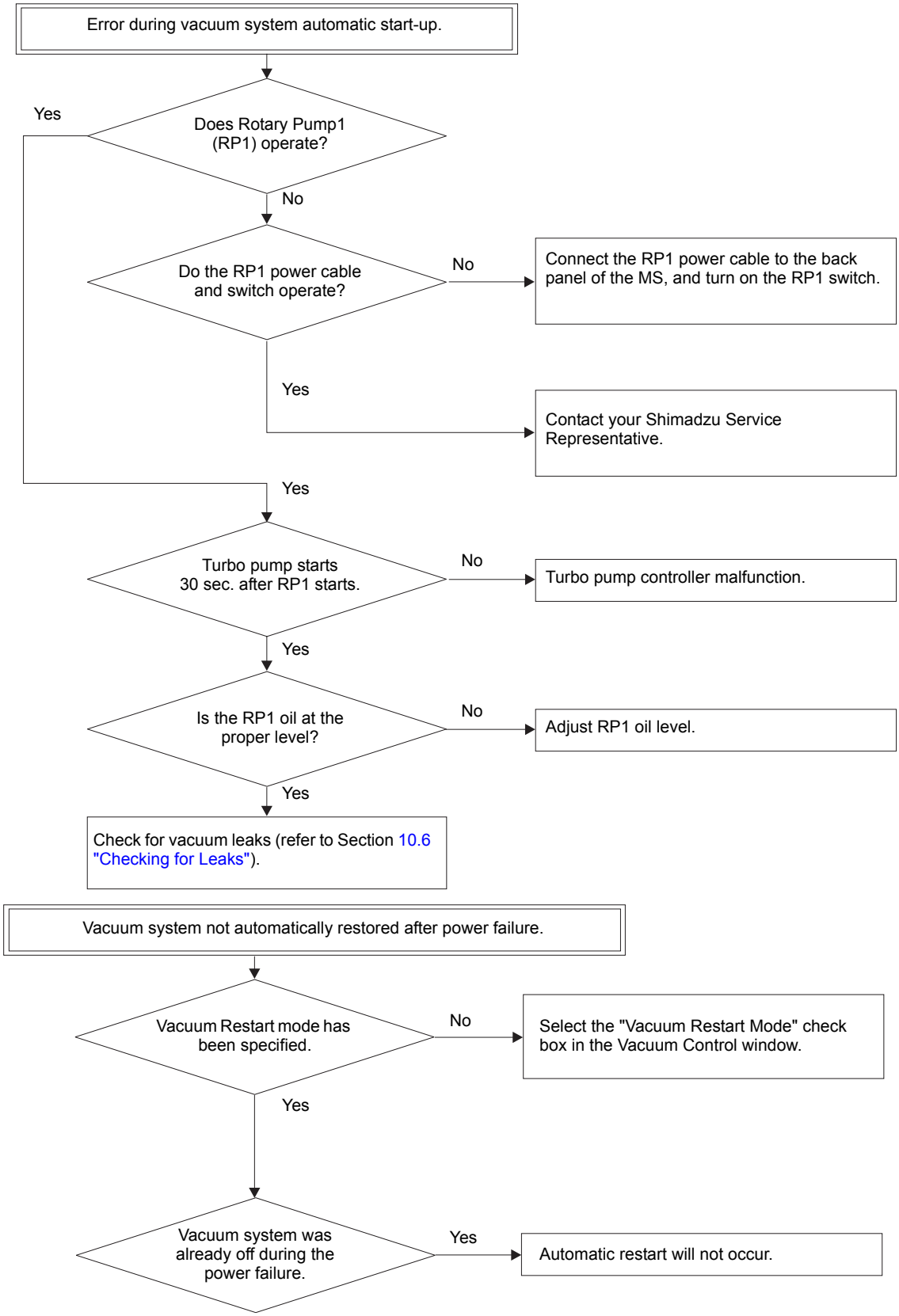


Figure 11.3 Vacuum System Startup Errors

11.1.4 MS Filament ON Errors

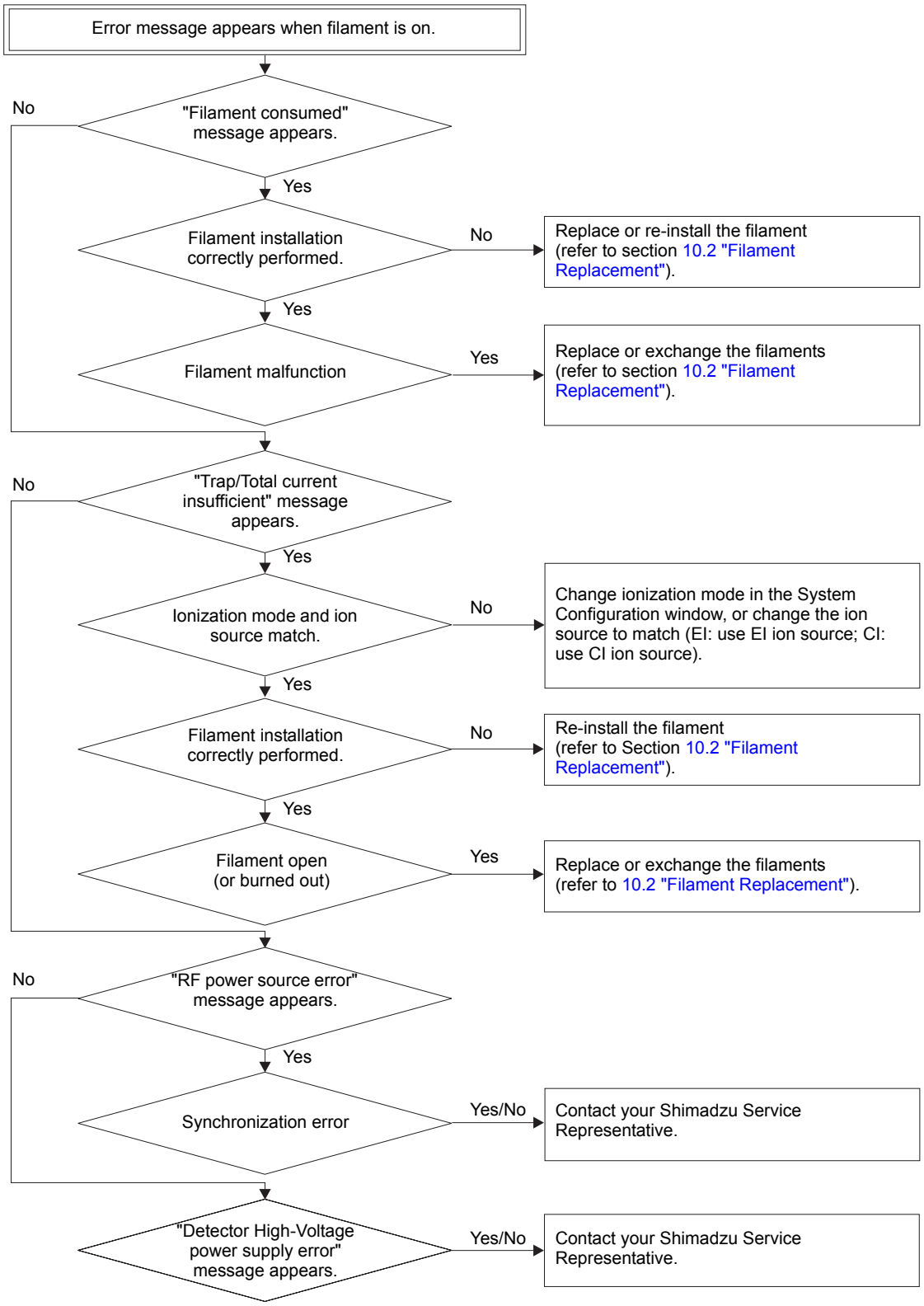


Figure 11.4 MS Filament ON Errors

11.1.5 Autotuning and Analysis Errors

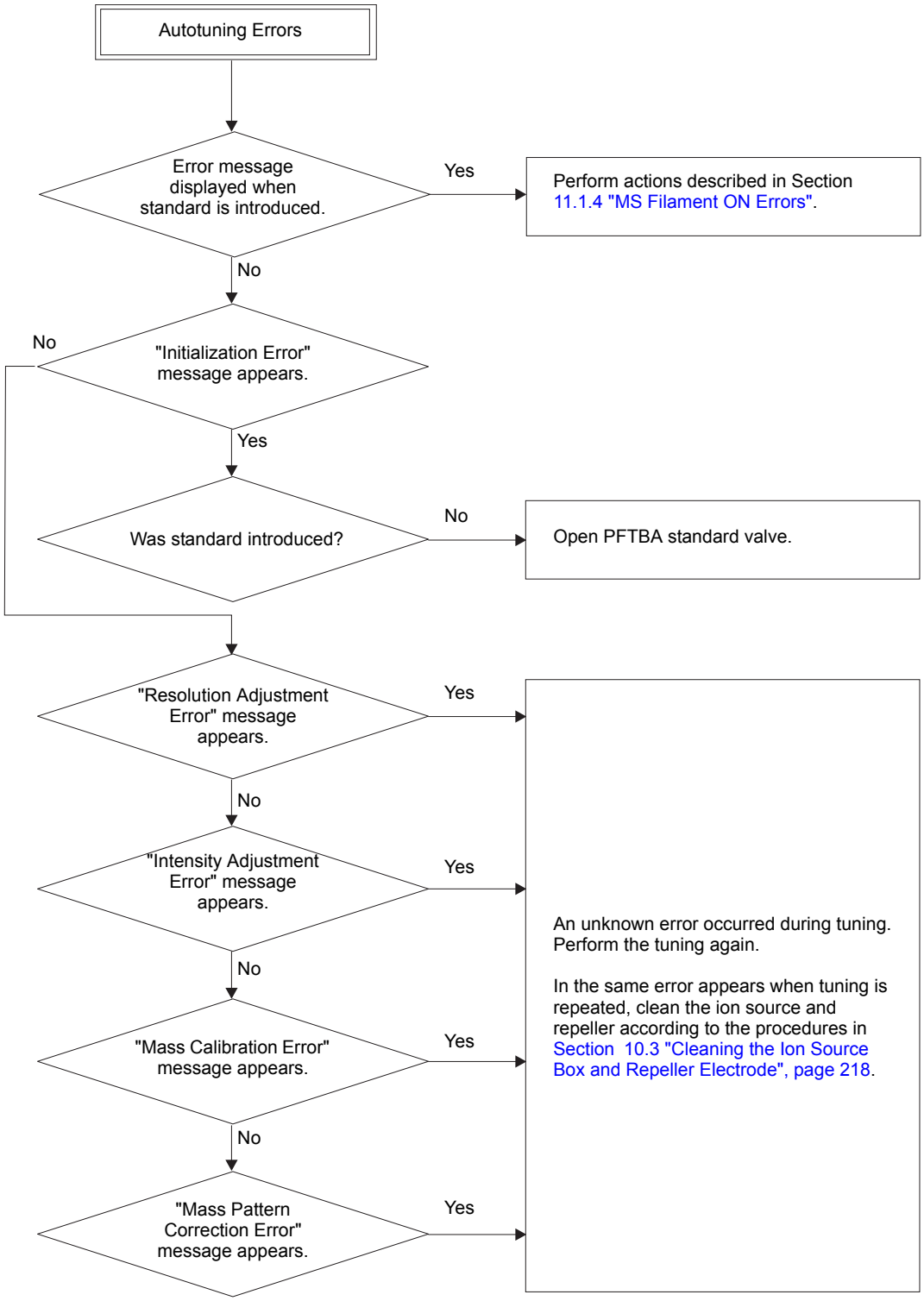
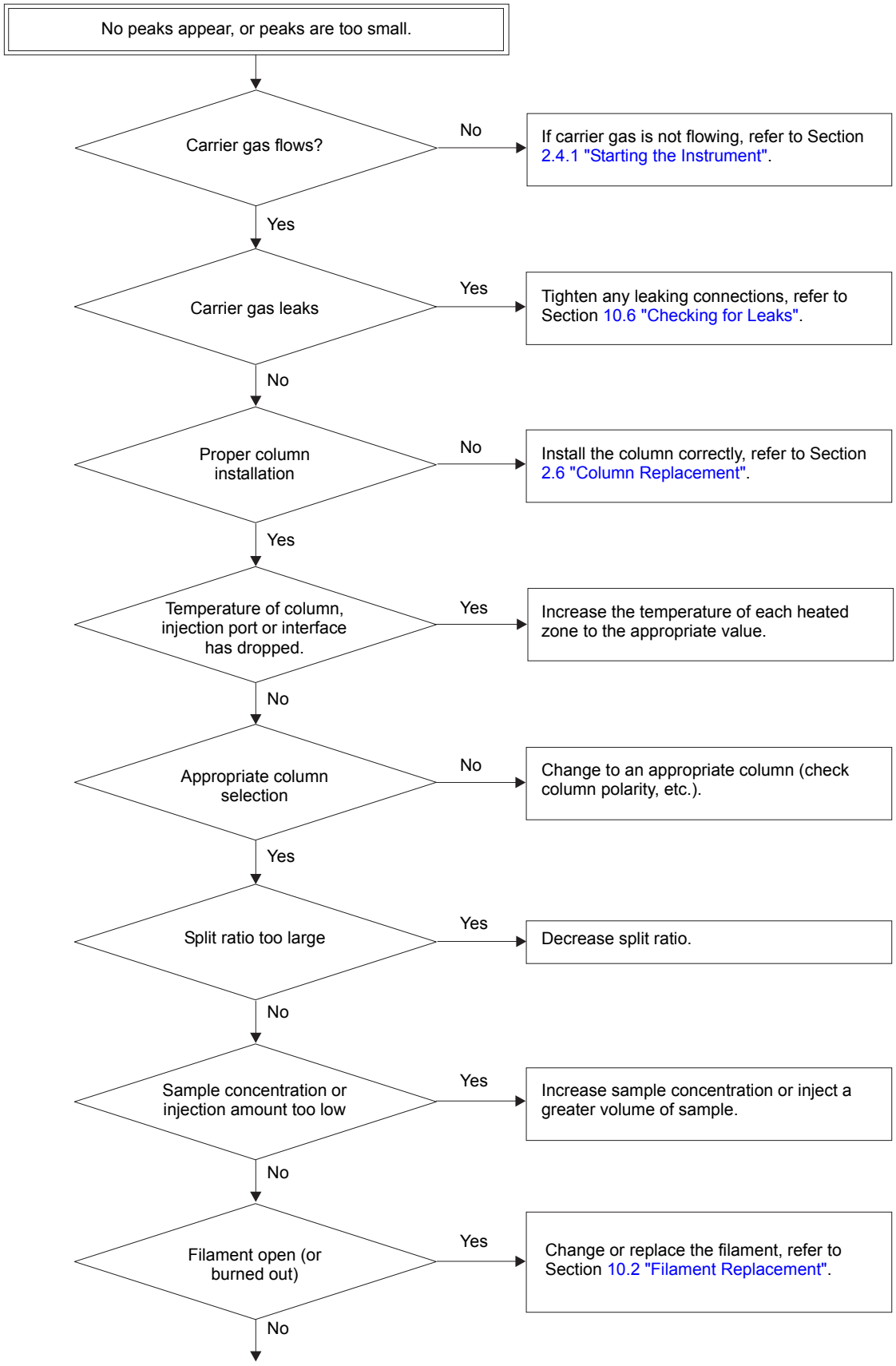


Figure 11.5 Autotuning Errors


No peaks appear, or peaks are too small.
Carrier gas flows?
No
If carrier gas is not flowing, refer to Section 2.4.1 "Starting the Instrument".
Yes
Carrier gas leaks
Yes
Tighten any leaking connections, refer to Section 10.6 "Checking for Leaks".
No
Proper column installation
No
Install the column correctly, refer to Section 2.6 "Column Replacement".
Yes
Temperature of column, injection port or interface has dropped.
Yes
Increase the temperature of each heated zone to the appropriate value.
No
Appropriate column selection
No
Change to an appropriate column (check column polarity, etc.).
Yes
Split ratio too large
Yes
Decrease split ratio.
No
Sample concentration or injection amount too low
Yes
Increase sample concentration or inject a greater volume of sample.
No
Filament open (or burned out)
Yes
Change or replace the filament, refer to Section 10.2 "Filament Replacement".
No

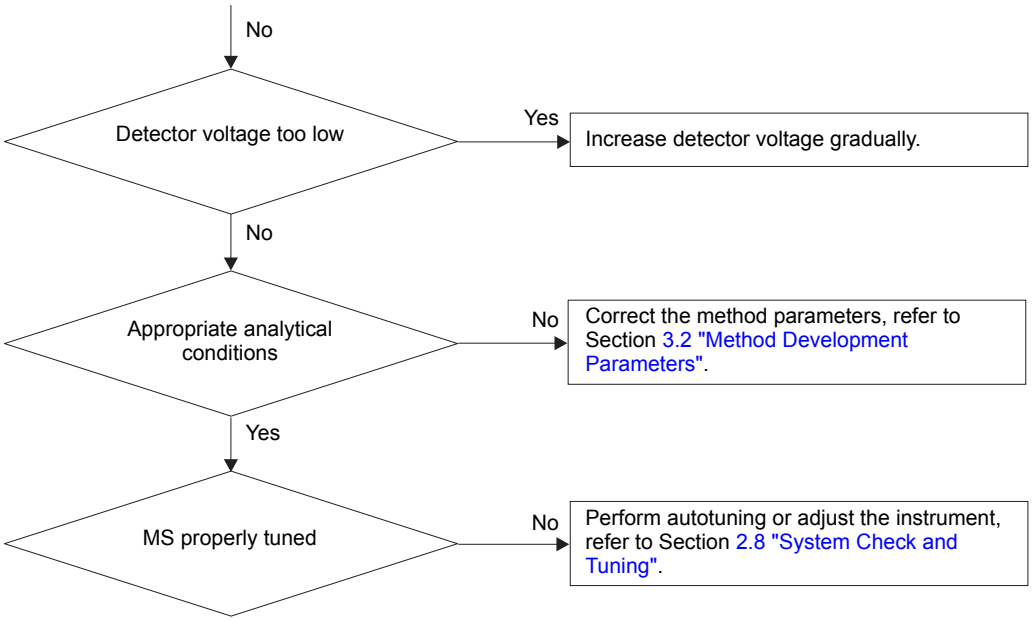


Figure 11.6 No Peaks or Small Peaks

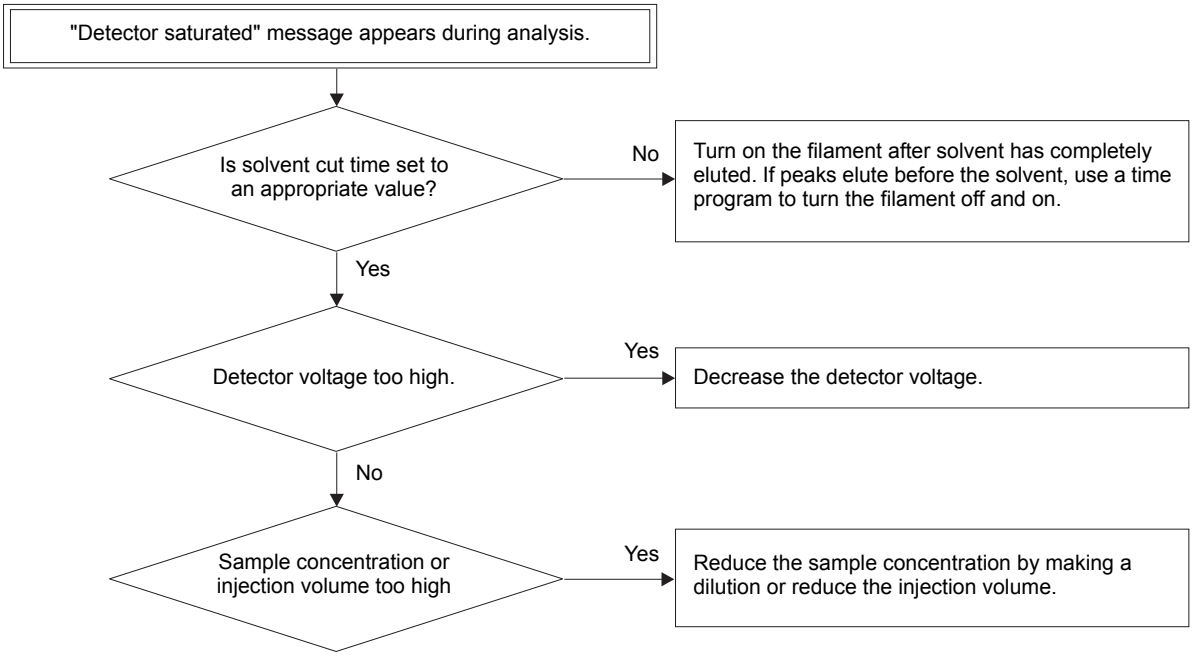


Figure 11.7 Analysis Errors

11.2 Vacuum System Protection Functions

This section describes the protection functions when something abnormal is detected in the vacuum system.

11.2.1 Overview

Three items are monitored during evacuation in the GCMS-QP2010. System protection occurs when something abnormal is detected.

- Status of the turbomolecular pump.
- Back pressure of the turbomolecular pump. (Low vacuum in "Instrument Monitor" shows the backpressure)
- Vacuum of analyzer housing. (High vacuum in "Instrument Monitor" shows the vacuum)
    - When the Ion Gauge is set to "None" in the System Configuration window, high vacuum is not displayed.
    - In case of Single TMP model, high vacuum is not monitored.

11.2.2 Protection Functionality

1. The filament, detector high voltage and RF power supply are turned off when the following occur:

(Dual TMP model)
(1) Low vacuum (monitored by Pirani gauge) is greater than 100.0 Pa.
(2) Turbomolecular pump is not in "Ready" condition.
(3) High vacuum (monitored by ionization gauge) is larger than 1.0E-2 Pa.

(Single TMP model)
(1) Low vacuum (monitored by Pirani gauge) is greater than 25.0 Pa.
(2) Turbomolecular pump is not in "Ready" condition.

Filament, detector high voltage and RF voltage are turned on again when the following conditions are satisfied.

(Dual TMP model)
(1) Low vacuum (monitored by Pirani gauge) is smaller than 80.0 Pa.
(2) Turbomolecular pump is in "Ready" condition.
(3) High vacuum (monitored by ionization gauge) is smaller than 0.8E-2 Pa.

(Single TMP model)
(1) Low vacuum (monitored by Pirani gauge) is less than 20.0 Pa.
(2) Turbomolecular pump is in "Ready" condition.

2. The vacuum system is automatically stopped when one or more of the following condition continue for more than 5 minutes.
(1) Low vacuum (monitored by pirani gauge) is larger than 100.0 Pa.
(2) Turbomolecular pump is not in "Ready" condition.
The vacuum system LED is red and blinks when the vacuum system is stopped.

Appendix A Peak Processing and Mass Spectrum Operations

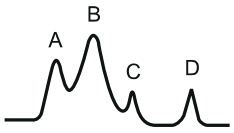
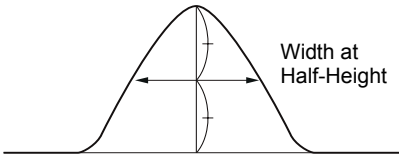
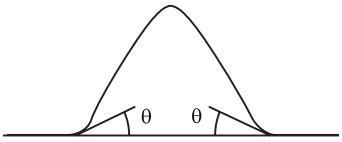
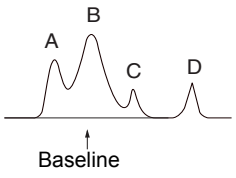
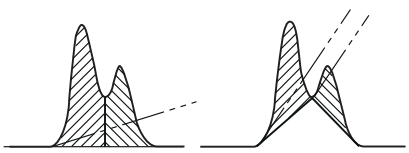
A.1 Integration and Peak Processing Parameters

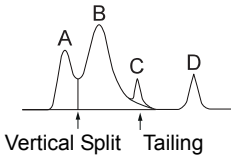
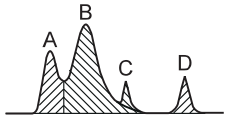
This section describes the peak detection algorithms and explains the types of peak processing that can be performed. The described parameters can be used in combination to properly detect and identify peaks.

A.1.1 Integration Parameters

In the table below, peak processing parameters are described in the order in which processing occurs. A setting range is given where applicable. Default values are shown in parenthesis.

Normally, the default values for the parameters can be used to detect peaks properly. For complex chromatograms, determine optimal values by referring to Section A.5.1 "Peak Processing", page 304.

Peak Processing Order	Parameter	Function Description Range (Default)	Comments
Peak detection 	Width	Minimum peak width 0.04 - 200 sec (2.0 sec)	Set to the width at half-height of the narrowest peak occurring during analysis. 
	Slope	Peak detection sensitivity 0 - 4E+11 /min. (100.0 /min.)	Sensitivity of peak detection Slope = tan θ 
	Peak Processing mode	Auto (Area)	The slope is adjusted automatically so that the number of peaks specified is obtained. If the specified peak number is not reached after slope has been adjusted, peaks with the largest areas are included until the set number of peaks is reached.
		Auto (Height)	The slope is adjusted automatically so that the number of peaks specified is obtained. If the specified peak number is not reached after slope has been adjusted, the highest peaks are included until the set number of peaks is reached.
		Detail	Set the parameter. If peak processing is performed in the automatic mode, the final slope value can be verified in the spectrum process table.
	# of Peaks	Maximum number of detected peaks (5)	If peak processing is performed in the automatic mode, the number of peaks to be detected can be specified. For certain types of chromatograms, there may not be enough peaks to reach the specified number.
Baseline processing 	Drift	Magnitude of baseline fluctuation –1E+7 - 1E+7 /min (0.0 = automatic processing)	Distinguishes between a peak and baseline drift.  When Drift is set to 0, baseline correction is performed automatically.

Peak Processing Order	Parameter	Function Description Range (Default)	Comments
Processing peaks with poor separation  Measurement of peak area  	None	Automatic	If peaks are not separated sufficiently (co-eluting), they are normally divided by drawing a vertical line perpendicular to the baseline to the valley between the peaks. Depending on the width of the valley between the peaks, they may be divided by the baseline.
Other	T.DBL.	Width & slope change time 0 - 10000 min (1000 min)	At the specified T.DBL time, the peak width parameter is doubled and the peak detection sensitivity parameter is halved. Automatic processing is performed when this parameter is set to 0.
	Min. Area/ Height	Min. Area/Height 0 - 10^9 (0)	Peaks with areas (or heights) below this value will be excluded from a similarity search and quantitation.
	Smoothing Method	None	Smoothing not performed.
		Standard	Moving averaging performed
		Savitzky-Golay	Smoothing performed by Savitzky-Golay method.
	Smoothing width	Smoothing moving average time width 0 - 200 sec (0 sec)	For noisy chromatograms, peak detection may not be performed correctly under normal peak processing conditions. In these situations, applying a moving average can perform additional smoothing. This parameter specifies the range of time over which the averaging occurs.
		(3 - 25 point)	Use only odd numbers when setting the number of smoothing points using the Savitzky-Golay method.
	# of Smoothing Times	Smoothing repetitions 0 - 99 (0)	Smoothing effects can be increased for a given chromatogram by repeating the moving averaging procedure. This parameter specifies the number of repetitions.

A.1.2 Width

Width is the most important of the peak processing parameters for optimizing peak detection.

To set the Width, determine the narrowest peak that will be analyzed on the chromatogram. Use a value for Width less than the width of this peak at half-height. Peak detection occurs for peaks that are approximately 1/4 of the specified width.

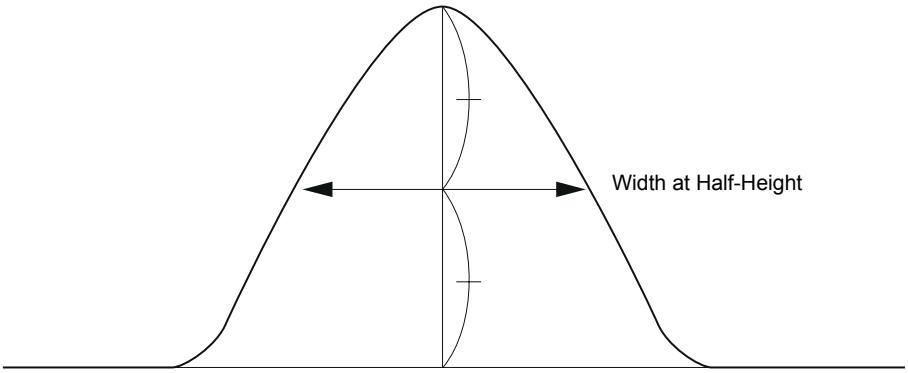


Figure A.1 Determining the Half-Width Value

Errors in peak detection and peak area determinations will occur if the Width is set incorrectly. The Width parameter significantly changes the way peaks are processed. Use a value that is appropriate for the narrowest peak to be analyzed to set this parameter.

Note

Removing unwanted peaks using Width
Noise peaks are generally narrower than target peaks. Using the width of the smallest target peak to determine the Width value will eliminate noise peaks from peak processing.

A.1.3 Slope

Peak detection is performed using the peak slope as shown in the diagram below. Peak slope is the linear slope of the chromatogram at peak start.

Peak start is determined when the peak slope becomes larger than the set value. Conversely, the peak end is determined when the slope becomes smaller than the negative of the set value.

The starting and end points are calculated from a quarter value of the Width parameter.

Slope is also known as peak detection sensitivity.

As the Slope value increases, the peak detection sensitivity decreases. Conversely, as the Slope value decreases, the peak detection sensitivity increases, allowing even broad peaks to be detected.

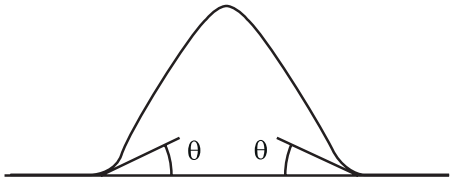


Figure A.2 Peak Detection and Slope

Note

Slope value criteria

The optimal slope value for peak processing depends on factors such as noise conditions and peak width. For capillary GC, the following values are generally observed.

Slope = maximum peak height/2000

For example, if the height of the largest peak is 146896 counts, then the Slope is set to a value of about 70 (/min).

The height of the largest peak can be easily obtained from the "GCMS Postrun Analysis" window or "Chromatogram Compare" window.

If too many unwanted peaks are detected, double the Slope value until the desired peak processing is obtained. Conversely, if the target peaks are not detected, halve the slope value until they are detected.

A.1.4 Drift

1. Automated baseline processing (Drift = 0)
Baseline correction will be performed automatically when the Drift parameter is set to 0. Two peaks are co-eluting when the width of the valley between the peaks (T2) is smaller than the estimated width of the immediately preceding peak at half height (T1). Peaks are separated by processing as described in the following section. If T2 is larger than T1 the baseline is adjusted to separate the two peaks.

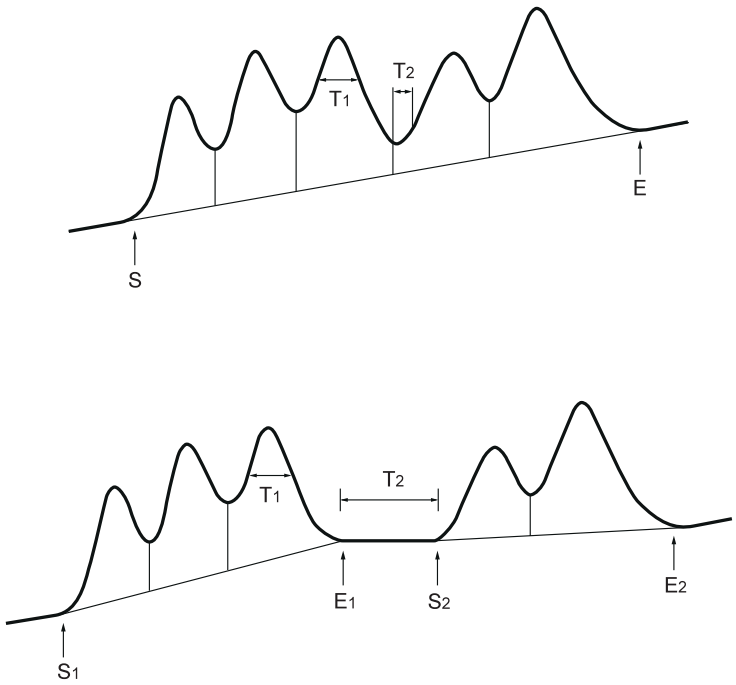


Figure A.3 Automatic Drift Processing with Narrow and Broad Valleys

2. Baseline processing for specified Drift values (Drift ≠ 0)
Specifying a value other than 0 for Drift in a time program will result in the region being processed by baseline correction, even in locations where the valley width (T2) is narrow.

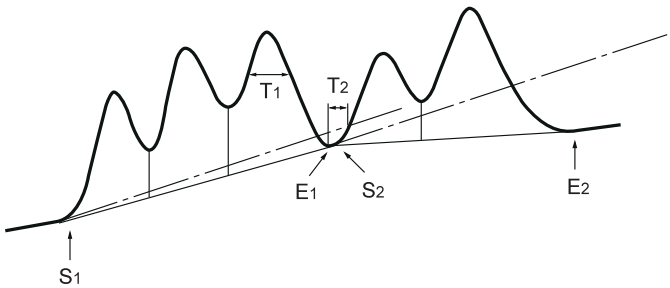
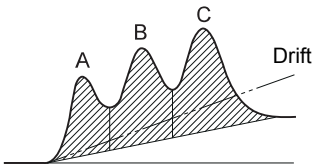


Figure A.4 Baseline Processing when Drift Is Set to a Value other than 0

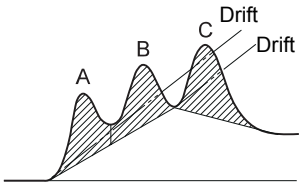
Baseline correction is performed differently from when the Drift is 0. The slope set with the Drift value extends along the dotted line shown in the diagram from the point of peak initiation (S). A corrected baseline is created when the peak end point is below the Drift slope line.

Note

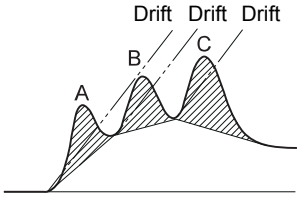
1. Changes in the Drift parameter will cause the baseline to be determined differently for identical chromatograms as shown below.



With a low drift setting, three peaks A, B and C are processed as co-eluting peaks.



A and B are processed as co-eluting peaks, and C is processed as a completely separate peak.



With a high drift setting, the peaks will be completely separated.

2. Set the Drift to a slightly higher value than the baseline drift for portions of the chromatogram where peaks appear.
   If the Drift setting is too low, the end points of peaks will fall below the Drift level, causing peaks to be analyzed as co-eluting.

A.1.5 Processing Co-Eluting Peaks

Two or more peaks that are inadequately separated when detected by the parameters of Width, Slope and Drift are considered to be co-eluting.

A determination is automatically made whether the co-eluting peaks are simply to be separated vertically or treated as a tailing peak with a smaller peak eluting in the tailing area.

Vertical Separation

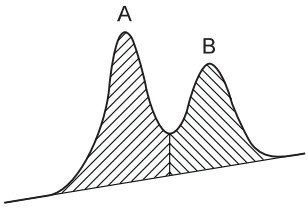
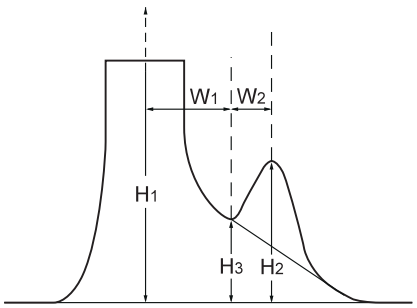


Figure A.5 Vertical Separation

Co-eluting peaks are generally processed by vertical separation.

Tailing Processing

The use of tailing processing is based on the two peaks' height ratio, the height of the valley and the separation conditions. The tailing peak and the peak present on the tail can then be determined.



Ratio of two peak heights H1/H2
Valley height H2/H3
Separation conditions W1/W

Figure A.6 Tailing Processing

Processing of Co-eluting Peaks

The automatic processing of co-eluting peaks is illustrated below.

Peak A is processed as tailing, and peaks B and C are processed as peaks present on the tail of the main peak A.

Co-eluting peaks that are present on the tail, such as peak C, are processed by vertical separation.

Peaks E, F and G are also co-eluting peaks, and are processed by vertical separation.

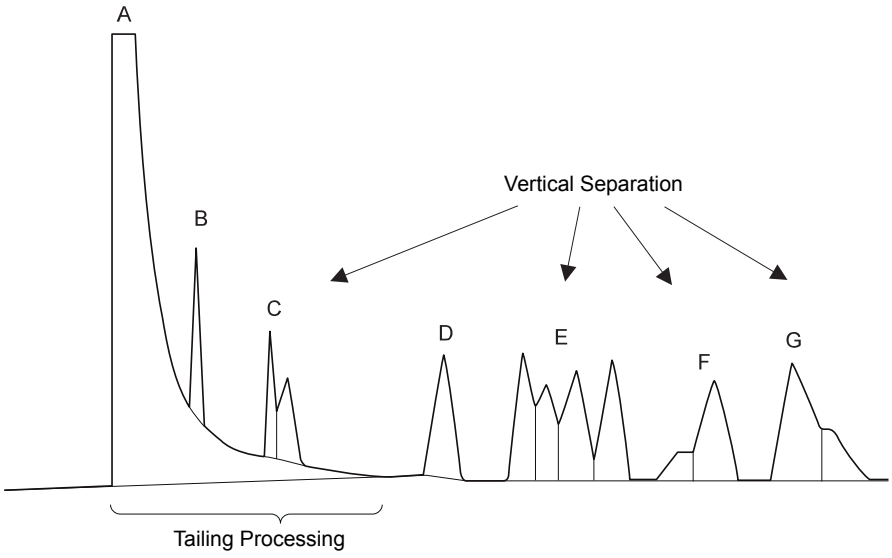


Figure A.7 Co-eluting Peaks Processed on a Tail and by Vertical Separation

A.1.6 T.DBL

1. Width and Slope values changed automatically (T.DBL = 0)
When T.DBL is set to 0, the Slope and Width parameters change automatically according to peak width. Peaks in isothermal GC analysis are initially narrow and their width increases over time. Early-eluting peaks have steep slopes, so the peak detection sensitivity (Slope) needs to be relatively low; later peaks are much broader and require increased peak detection sensitivity. The Width value must also be increased over time to detect broader peaks.

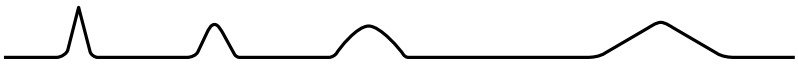


Figure A.8 Slope and Width Values Change Automatically when T.DBL = 0

A T.DBL setting of 0 (automatic) should not be used for chromatograms where the peak widths do not increase over time, as in GC temperature programmed analysis. This setting should also not be used when a broad peak appears long after a sharp peak, as in the next diagram, or when peak widths narrow after they were broad. In these cases, set T.DBL to a value other than 0.

Figure A.9 Use a T.DBL Value Other Than 0

2. When T.DBL is set to a value other than 0, the Slope (peak detection sensitivity) and Width (minimum width at peak half-height) parameters change by a factor of two, as shown below, at a time interval specified by T.DBL.

Automatic changes in Slope and Width using T.DBL

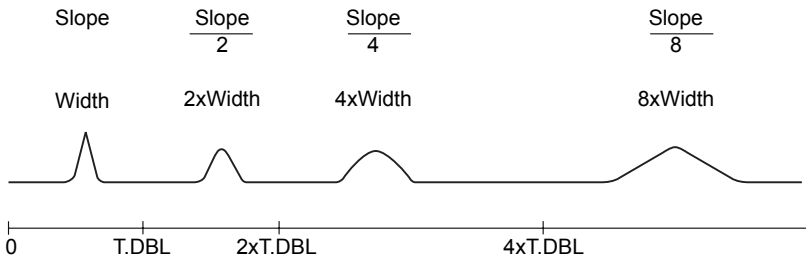


Figure A.10 Slope and Width Changes with T.DBL

Note

For qualitative processing, up to 15 Width changes are permitted per chromatogram. For quantitative processing, up to 2 changes are allowed, including Width or T.DBL changes in a time program. One chromatogram is counted per ID.

Normally, T.DBL is set to the time required for a peak to reach twice its initial peak width. If it is difficult to find a peak on the chromatogram that doubles in width, the value can be calculated as shown below.

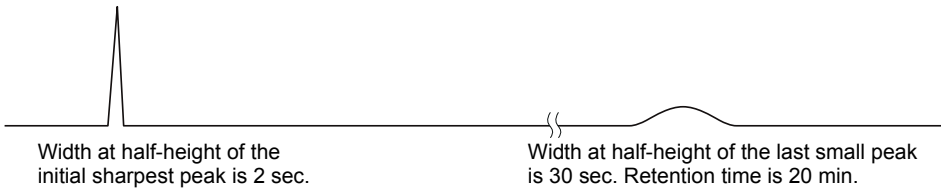


Figure A.11 Determining T.DBL when a Peak that Doubles in Width Cannot Be Located

(1) Use the width at half-height of the initial sharpest peak for the Width parameter. 2 sec is used in this example.
(2) Measure the retention time and width at half-height of the last small peak. A retention time of 20 min, and a width at half-height of 30 sec are used.
(3) Time required for 2x increase = (2 sec / 30 sec × 20 min × 2) = 2.7 min.
General formula:

$$\text{T.DBL} = \frac{\text{First Peak Width at Half-Height}}{\text{Last Peak Width at Half-Height}} \times \text{Last Peak Retention Time} \times 2$$

3. Disabling T.DBL so that Width and Slope values not changed automatically.
For most GC temperature-programmed analyses, peak widths do not increase over time and the Width and Slope values should not be changed automatically. To disable T.DBL, set it to a value longer than the final time for peak processing.

A.1.7 Peak Processing Time

Peak processing occurs only during the interval specified by the peak processing start and end times.

If peak processing ends as the peak slope is increasing, the area of that peak will not be determined.

If peak processing ends as the peak slope is decreasing, the peak area will be determined up to that time.

If peak processing ends as the peak slope is increasing for a co-eluting peak, the former peak is considered to be the final peak. In the following example, the calculated area is shaded.

Effect of end peak processing during a peak.

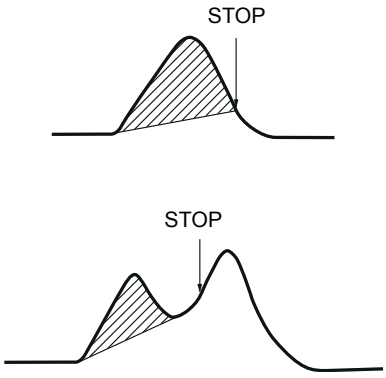


Figure A.12 Ending Peak Processing during a Peak

A.1.8 Peak Processing With a Time Program

Time programs can be used as part of a method to repeat the same processing for several chromatograms. A time program can be used as a peak processing method for routine analysis.

In addition to the peak processing parameters described above, a time program can be used to suspend peak processing during a specified time frame, removing unnecessary peaks, and to adjust tailing processing ranges. Peaks that were not processed as tailing during normal processing can be specified as tailing in a time program.

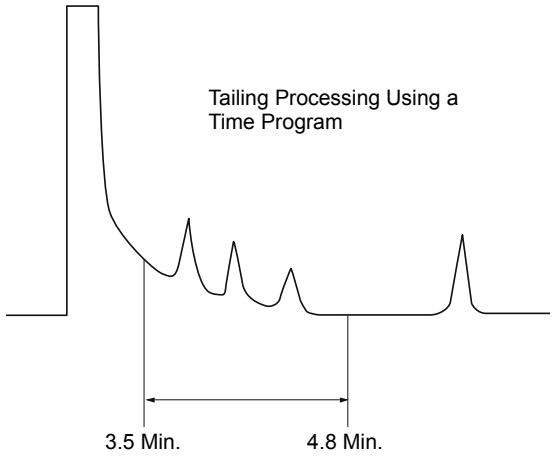


Figure A.13 Peak Processing Using a Time Program

A.1.9 Minimum Area/Height

This parameter has no direct effect on peak processing. After normal peak processing occurs based on the peak processing parameters, the minimum area/height values can be used to remove peaks from the following locations:

(1) Drawing the baseline, annotation and peak detection marks (display and report)
(2) Peak report
(3) Spectrum process table
(4) Quantitative (or calibration) calculations

A.1.10 Smoothing

For very noisy or complex chromatograms, or in situations where peak detection does not occur properly under normal peak processing conditions, applying a moving average can smooth the chromatogram.

Smoothing width: Sets the average width over which the moving average is taken, in units of seconds.

# of Smoothing Time: When a single moving average smoothing has little effect, the smoothing can be repeated several times. This enhances the effects of the moving average operation.

Note

Subjecting the actual chromatogram data to moving averaging performs smoothing. Consequently, the actual chromatogram is changed. The use of smoothing should only be considered when processing is not possible under ordinary peak processing conditions.

A.2 Mass Spectrum Operations

This section describes the two mass spectrum editing functions, Averaging and Background Subtraction. These functions allow the extracting of complete spectrum information under various conditions.

A.2.1 Mass Spectrum Background Correction (Subtraction)

Data obtained by mass spectral measurement will contain peaks associated with column background and residual gases. A high degree of separation can be obtained with a capillary column, but some peaks cannot be completely resolved. In these instances, the peaks of adjacent compounds overlap with those of the target compounds, making qualitative processing difficult. More accurate qualitative processing may be accomplished by applying background subtraction to the mass spectrum.

Background subtraction is performed as follows. First, the masses of all of the mass peaks appearing in the two spectra for the target and background are rounded to obtain integer masses. Next, the intensities of the same integer masses appearing in the target and background spectra are subtracted and the masses including decimal places, are returned to the masses in the original target spectrum. Resulting intensities that have a negative value are omitted at this time.

A.2.2 Mass Spectrum Averaging

When MS analysis is performed in scan mode, a spectrum scan is performed linearly from the low to the high masses. This can favor the high mass relative abundances or favor the lower mass relative abundances, depending on when the peak elutes relative to the acquired scan.

For example, when using a 0.5 second scan and a mass range of 10amu to 700amu, the time from when the intensity of the mass 10 is analyzed to the time when the intensity of the mass 700 is analyzed is 0.5 seconds. Spectra are different depending on the exact point in time that the peak's true apex elutes compared to the start of the scan. The ratios of low mass intensities and high mass intensities will be different in terms of the analyzed spectrum versus the actual spectrum. Averaging throughout the mass spectrum, including the peak apex can produce a more accurate mass spectrum.

The process of mass spectrum averaging is used to compensate for the relative abundance distortion seen on the leading side and on the tailing side of chromatographic peaks as well as at the apex. Either the scans of the entire peak can be averaged or an equal number of scans from the front side and tailing side should be averaged with the apex. This gives an averaged spectrum that represents the true relative abundances of the ion fragments that existed in the ion source and traveled to the detector.

Mass spectrum averaging is performed as follows. First, the masses of all of the mass peaks that appear over the entire mass spectrum are rounded to integer masses. Next, the intensities of the integer masses are summed, and divided by the number of scans used for averaging. After averaging, the integer masses are given the exact mass assignments of the spectrum scan containing the greatest intensity.

A.2.3 Performing Mass Spectrum Operations and Similarity Searches During Automated Processing

TIC peak processing is automatically performed according to the Peak Integration and Spectrum Process parameters specified in the "Qualitative Parameters" dialog box. Detected peaks are subjected to averaging or background subtraction accordingly, and the spectrum is edited. The results are saved in the spectrum process table, and can be checked in the "Data Analysis" window. The spectrum can be averaged from peak start to peak end or at the peak apex. Select the background spectrum for subtraction from the drop-down list, which includes the peak starting or ending spectrum, or a spectrum obtained by averaging over any desired time interval.

An automated similarity search is performed on the edited spectra listed in the spectrum process table. If both qualitative peak processing and similarity searches are specified, peak processing is first performed, followed by spectrum editing and similarity searches. If only similarity searches are specified, similarity searches can be performed automatically on edited spectra in the spectrum process table from the "Data Analysis" window.

If similarity searching is specified in the Batch Table in the "GCMS Real Time Analysis" window, then similarity searches and sample analyses are performed simultaneously. Because the results of peak processing and the number of detected peaks are not known beforehand, processing can be slow. It is better to perform automated similarity searches by reprocessing acquired data with a batch table in the "Data Analysis" window.

A.3 Similarity Search Parameters and Functions

This section describes the method for calculating the degree of similarity between spectra during a similarity search; pre-search parameters which speed up detection from a large library; and the post-search parameters.

Retention times are used to identify target components from a two-dimensional chromatogram. First, a standard is analyzed; then the retention times of the unknown peaks are compared to the retention times of the target peaks in the standard. This identification method is not always reliable, because other peaks can elute at the same time as the target peaks, especially in complex samples.

Three-dimensional spectral data obtained by GC/MS analysis, allows the identification of target components using a similarity search based on spectral information. For the similarity search, either a private library, produced from standard spectra, or a public library, supplied by NIST, Wiley, etc., can be used.

A.3.1 Similarity Search Calculations

The similarity index (degree of similarity) is a quantitative expression of the difference between the spectrum of an unknown sample and a spectrum recorded in a library. The difference in intensity of each of the spectral peaks at a given mass (m/z) is determined. The degree of similarity increases as the difference between the values decreases.

The similarity index (SI) is calculated using the equation below.

$$SI = \left(\frac{\sum_{m/z} |Iu(m/z) - It(m/z)|}{\sum_{m/z} \{Iu(m/z) + It(m/z)\}} \right)$$

Iu(m/z): Relative spectrum intensity of the m/z of the unknown mass spectrum.

It(m/z): Relative spectrum intensity of the m/z of a mass spectrum recorded in a library.

An SI of 100 indicates mass spectra that are identical, while an SI of 0 indicates spectra that are completely different.

A.3.2 Pre-Search Function

Frequently, the number of compounds registered in a public library is so vast that it is best to narrow the search with a pre-search. Pre-searching involves a simple comparison of the unknown sample spectrum to the spectra registered in the library. Library spectra are excluded from the search if there are clear differences between the spectra. This function acts, therefore, as a filter.

Certain masses commonly appear, while others rarely appear. The pre-search function has been based on the study of many mass spectra, determining which mass peaks are produced by fragmentation of the most types of molecules. A number is assigned based on the frequency of mass peaks for all masses. This information and the mass peak intensities are used to obtain the masses of the most characteristic peaks of the unknown mass spectrum. Pre-searching determines whether the ions for these masses are present in a spectrum registered in the library.

There are mass spectrum peaks for masses that rarely appear. If the peaks of these masses are above a certain intensity, the mass spectrum peaks are taken as the most characteristic peaks of an unknown spectrum. This information is used to create the list of compounds processed by the similarity search algorithm.

A.3.3 Similarity Search Parameters

There are three parameters used during the actual search: minimum similarity, search depth and hit number.

As explained above, the similarity index quantitatively expresses the differences between a spectrum registered in the library and the spectrum of an unknown compound. Since there is no interest in compounds that show very little similarity, library compounds with a similarity index below a certain value will be excluded from the search results.

The "search depth" sets the range of a pre search. A search depth of 1 limits the research to the most and next most characteristic spectrum peaks. A search depth of 2 performs the research with respect to the most characteristic spectrum peak and the 2nd and 3rd most characteristic spectrum peaks. As the search depth increases, the amount of data considered during the research increases, along with the similarity calculations on registered library spectra. The pre-search effectiveness as a filter diminishes; resulting in a broad search which requires more time to complete.

Normally, a search depth of 1 is sufficient. However, if the calculated similarity index is extremely low, or when performing a library search for a mass spectrum with a high impurity content, a greater search depth may be more appropriate.

The hit numbers obtained from spectra with the greatest calculated similarities are displayed in order in the "Similarity Search Results" window.

A.3.4 Post-Search Functions

A post-search can be conducted when the molecular weight or number of carbon atoms of the unknown compound is fairly well known. For example, if the molecular weight of the component is known, the hit list is produced from only the compounds within that molecular weight range.

The parameters that are used for post searching are: molecular weight, compound name, structural formula, class flag, retention index and base peak. Post-searches can be performed using these six parameters in combination.

Retention Index
The absolute value of the peak retention time changes according to the analytical conditions. Therefore, the elution order of a specific compound can be more universally expressed by taking the proportion of the retention time of the specific compound to the retention time of a standard peak. That ratio is called the retention index (Ix) and is found by the following formula.

Isothermal analysis:

$$Ix = \frac{\log\{(t_x - t_m)/(t_n - t_m)\}}{\log\{(t_{n+1} - t_m)/(t_n - t_m)\}} \times 100 + (n \times 100)$$

Temperature-programmed analysis:

$$Ix = \frac{(t_x - t_n)}{(t_{n+1} - t_n)}$$

Where:

t_x: Retention time of compound to be analyzed

t_n: Retention time of the n-paraffin peak with a carbon number of n eluting prior to the compound to be detected

t_{n+1}: Retention time of the n-paraffin peak of carbon number n+1 eluted after the compound to be detected

t_m: Methane retention time

A.4 Peak Identification, Quantitative Calculations, and Calibration

There are three quantitation processing functions directly associated with quantitation: Peak Identification, Quantitation and Calibration. This section describes these three functions and the ID numbers assigned to each component.

A.4.1 ID Numbers

Identification, calibration and quantitation parameters must be specified for each compound to perform quantitation of compounds separated by GC. The ID number is the number assigned to each component so that the parameters listed above can be specified individually.

Although the ID numbers do not have to follow the peak elution order, doing this makes working with compound information much easier.

A.4.2 Peak Identification

When peaks are processed based on a Compound Table, peak identification is used to determine which peaks are target peaks. This determination is based on the peak retention time and the intensity ratio(s) of the target ion to the reference ion(s). A peak is identified as a target compound when both identification criteria are met.

1. Identification based on retention time

(1) Absolute retention time method

A target peak is identified based on the standard retention time and its retention time window or band, previously entered in the Compound Table. The actual compound retention time is used and identification is performed according to the conditional formula below.

$$\left| \begin{matrix} \text{Target Peak} \\ \text{Standard R. T.} \end{matrix} - \begin{matrix} \text{Target Peak} \\ \text{Actual R. T.} \end{matrix} \right| < \text{Target Peak R. T. window or band}$$

Note

When numerous peaks fall within the identification range, the peak that is closest to the standard retention time is identified as the target component.

(2) Relative retention time method

With the relative retention time method, identification is performed after correcting the retention time drift for each peak. First, a specified reference peak is identified by the absolute retention time method; then the target peaks are identified by the conditional formula below.

$$\left| \begin{pmatrix} \text{Std. R. T. of peak} \\ \text{to be identified} \end{pmatrix} - \frac{\begin{pmatrix} \text{Std. R. T. of the} \\ \text{reference peak} \end{pmatrix}}{\begin{pmatrix} \text{Actual R. T. of the} \\ \text{reference peak} \end{pmatrix}} \times \begin{pmatrix} \text{Actual R. T. of peak} \\ \text{to be identified} \end{pmatrix} \right| < \begin{pmatrix} \text{R. T. window or band for the} \\ \text{peak to be identified} \end{pmatrix}$$

The actual retention time of the target peak is corrected by calculating a ratio based on the shift in retention time of the reference peak. The corrected retention time is used for identification.

Since the TIC is used, the mass cannot be used to identify the components. This method is most effective when several peaks co-elute. The reference peak must be a distinct peak that does not co-elute.

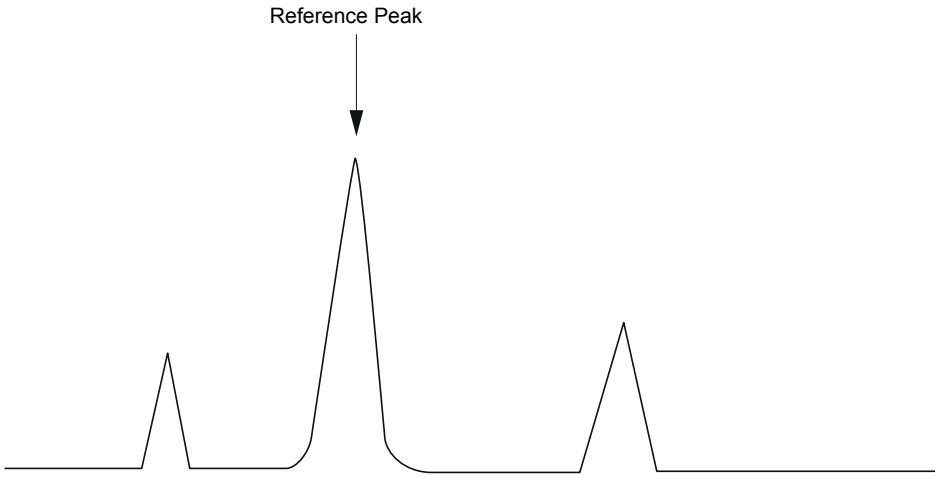


Figure A.14 Relative Retention Time Method

(3) Multi-reference relative retention time method

For capillary GC temperature-programmed analyses, the retention time shift is often not proportional to the retention time. Retention time correction for large retention time shifts can be improved by selecting multiple reference peaks. The peaks are classified into multiple zones and a reference peak is specified for each zone. The peaks within the zone are identified based on each zone's reference peak.
Peaks that elute prior to the first reference peak are identified by a simple relative retention time method using only the first reference peak. Subsequent peaks are identified with a corrected retention time, which is based on the retention time shift of the preceding and following reference peaks. Refer to the formula below.

$$\text{Target peak corrected for R. T.} = \frac{t - t_1}{t_2 - t_1} \times (T_2 - T_1) + T_1$$

Where:

t: Actual retention time of the target peak
t_1: Actual retention time of reference peak 1
t_2: Actual retention time of reference peak 2
T_1: Expected retention time of reference peak 1
T_2: Expected retention time of reference peak 2

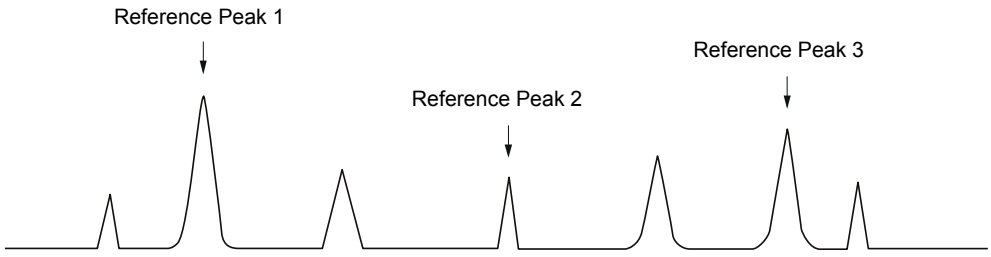


Figure A.15 Multi-Reference Relative Retention Time Method

(4) Window method

The retention time window method specifies a certain retention time span for the peaks. This window increases with retention time, as shown in the diagram below. For GC isothermal analysis, for example, it is often necessary to increase the retention time window over time. The disadvantage of this method is that the time span allowed for each component can overlap with the windows of other compounds.

$$\text{Retention time window (min)} = \frac{\text{Std. R. T. (min)} \times \text{Window (\%)}}{100} \times 0.02$$

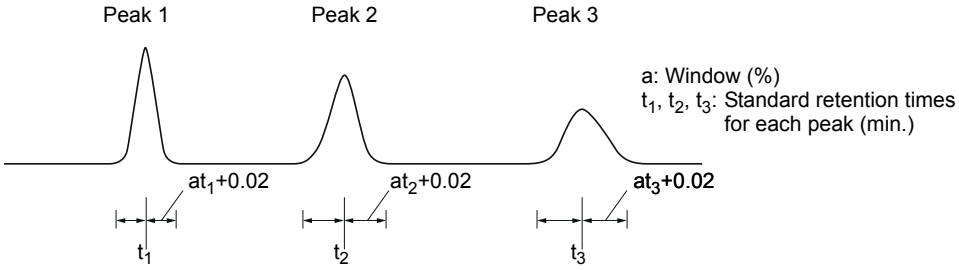


Figure A.16 Window Method

(5) Band method

The band method uses a specific retention time span for each peak. The advantage of this method is that an optimal retention time band can be specified for each peak. This method can be time-consuming, as it is necessary to make the setting for each peak.

The band method is useful when peak widths do not change over time, as in a GC temperature-programmed analysis.

Allowed time span (min) = Band (min)

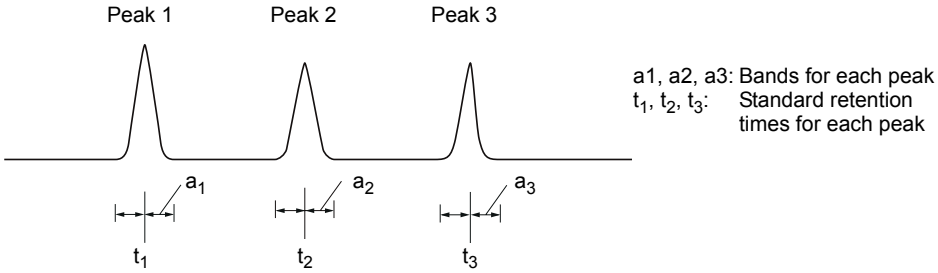


Figure A.17 Band Method

2. Identification by ion intensity ratio

In mass spectrum analysis, the relative intensity of each ion fragment is virtually constant and produces a value specific to each target compound, providing the analysis conditions are constant.

During an analysis, numerous ions are simultaneously analyzed, and their intensity ratios (peak area ratios or height ratios) are determined, allowing component identification. Peaks can be accurately identified using ion intensity ratios along with retention time. This avoids the mistaken identification of impurities with similar retention times.

For quantitative analysis, a maximum of five reference ions can be specified in addition to the target ion. After retention time identification, the components are re-checked for accurate identification by determining if the area or height ratios are within the specified ranges.

The reference ion intensity is specified as a percentage with respect to the intensity of the target ion.

Absolute Tolerance and Relative Tolerance Ranges

Reference ion spectrum confirmation can be absolute or relative. An absolute tolerance specifies the ratio with respect to the intensity of the target ion, regardless of the reference ion intensity. A relative tolerance specifies the ratio with respect to the intensity of the reference ion.

An absolute tolerance is usually sufficient, but in cases where the intensity of the reference ion is extremely small, and that ion is crucial to the identification of the peak, the accuracy of identification may be increased by using a relative tolerance.

Absolute tolerance range (%): Ri ± Rw

Relative tolerance range (%): Ri ± Ri • Rw/100

Ri: Ref. intensity ratio with respect to target ion (%)

Rw: Tolerance range (%)

3. Identification using pattern matching

Once a standard spectrum is registered in the compound table, a similarity calculation is conducted for spectra of detected peaks, and a compound is identified as the target compound if the similarity value exceeds the set minimum similarity value.

4. Grouping

By grouping such compounds as the isomer or homolog that have the similar characteristics, determining the concentration according to group is referred to as "grouping".

This software allows selection between two methods of grouping in the "Quantitation Parameters", as follows.

(1) Group Calibration

In grouping associated with Group Calibration, the peak areas or peak heights of compounds that have been grouped are summed, a calibration curve is drawn for each group and quantitation is conducted for each group as a whole.
The group area/height is as follows.
Group area/height = sum total of all compounds in the group

(2) Concentration Sum

In grouping associated with Concentration Sum, a calibration curve is drawn for each compound, and after quantitation is conducted for each compound, the sum of the concentrations of the compounds in the group is taken as the group concentration.

A.4.3 Quantitative Calculation Methods

Quantitation is possible by six different methods.

Area Normalization

The detected peak areas or heights are totaled, and the percentage of each peak area or height is determined with respect to the total value.

$$\text{Concentration (\%)} = \frac{A_i}{\sum A_i} \times 100$$

Corrected Area Normalization

This method uses the external standard calibration curve method to quantitate each peak. The quantitation values are totaled and the percentage of the quantitation values for each component are determined with respect to the total.

$$\text{Concentration (\%)} = \frac{(A_i - F2_i)/(F1_i)}{\sum\left\{\frac{(A_i - F2_i)}{F1_i}\right\}} \times 100$$

Internal Standard

An internal standard compound is added to each standard and sample. Then, a calibration curve is created, expressing the relative sensitivity and mass ratio of a standard target peak relative to the internal standard peak. The quantitation value of the target compound is then calculated by applying this calibration curve to the area ratio or height ratio of the unknown peak. This method takes into account variations in analytical conditions.

$$\text{Concentration} = \frac{A_i/(A_{IS} - F2_i)}{F1_i} \times \frac{W_{IS}}{W_{spl}} \times DF$$

External Standard (Absolute Calibration Curve Method)

This method determines the concentration of a target compound by creating a calibration curve from the relationship between the absolute mass or concentration of a compound in a standard and the area or height of its peak. An unknown sample is analyzed under identical conditions, and then the calibration curve is applied to the area or height of the peak in the unknown. The analytical conditions of the unknown sample must be exactly the same as those of the standard. Since the method accuracy is dependent on the volume of the sample injected injection volumes must be constant.

$$\text{Concentration} = \frac{A_i - F2_i}{F1_i} \times \frac{DF}{W_{spl}}$$

Corrected Area Normalization with Scale Factor

This method calculates peak concentrations using the total area or height as a scale factor (dilution factor), rather than letting the total be 100.

$$\text{Concentration} = \frac{(A_i - F2_i)/(F1_i)}{\sum\left\{\frac{(A_i - F2_i)}{F1_i}\right\}} \times SF$$

Where:

A_i: Peak area (height)

$F1_i$: Slope correction factor

$F2_i$: Constant (intercept)

W_{spl}: Sample amount

W_{IS}: Internal standard amount

DF: Dilution factor

SF: Scale factor (use dilution factor value)

Standard Addition

The matrix effect refers to coexisting compounds biasing the concentrations calculated for the target compound. The standard addition method counters this problem by adding several concentrations of a known test compound to identical quantities of sample and calculating the concentrations of the samples. This is accomplished by preparing several vials of sample; one vial is left unspiked and the others are spiked with various concentrations of standard. After analysis, a calibration curve is created with the quantity of standard added on the horizontal axis, and the peak area or height on the vertical axis. Quantitation of the sample is performed using this calibration curve.

A.4.4 Calibration

By analyzing a known concentration of standard, a calibration curve can be created and used for quantitation.

The horizontal axis of the calibration curve represents the amount of component (ratio with respect to the internal standard when an internal standard is used). The vertical axis represents area or height (ratio with respect to the internal standard peak when and internal standard is used). A linear or point-to-point curve is drawn.

The calibration curves can be made to pass through the origin, if desired (except for the Average RF curve, which must pass through the origin).

Calibration curves are classified into four types.

Linear
Up to 64 different standard concentrations are analyzed, and a linear calibration curve is calculated by the least squares method.

If there is only a single calibration level, a simple straight line passes through the single point and the origin.

If there are two or more calibration levels, and the curve does not pass through the origin, a linear calibration curve is drawn through these two points.

In other cases, a linear curve is drawn using the least squares method.

Each point can represent the average of up to 10 individual analyses.

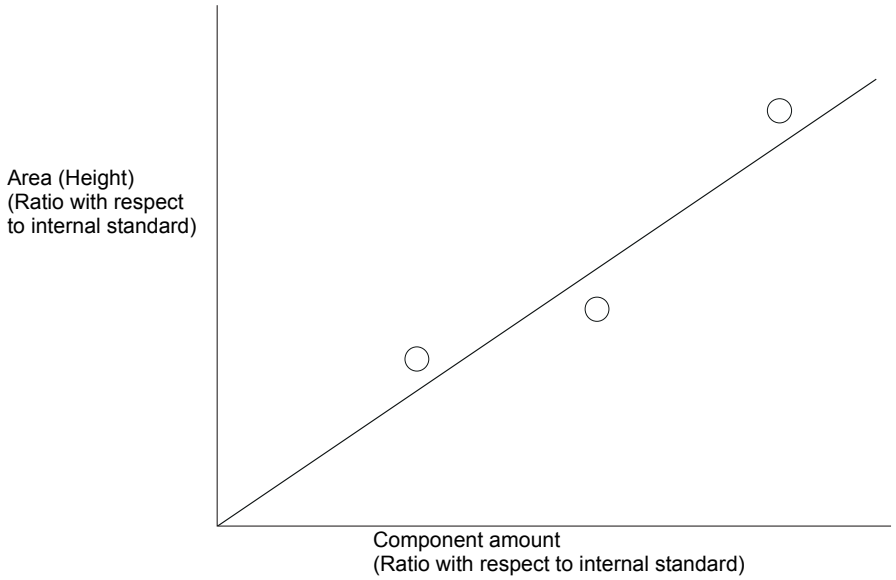


Figure A.18 Linear Calibration Curve

Quadratic/Cubic

Up to 64 different standard concentrations are analyzed, and a second order (quadratic) or third order (cubic) curve is drawn by the least squares method.

For quadratic curves, three or more calibration points are required. The curve will be calculated as linear if there are two or fewer points.

For cubic curves, four or more calibration points are required. The curve will be calculated as a quadratic equation if there are three points and as linear if there are two or fewer points.

Each point can represent the average of up to 10 individual analyses.

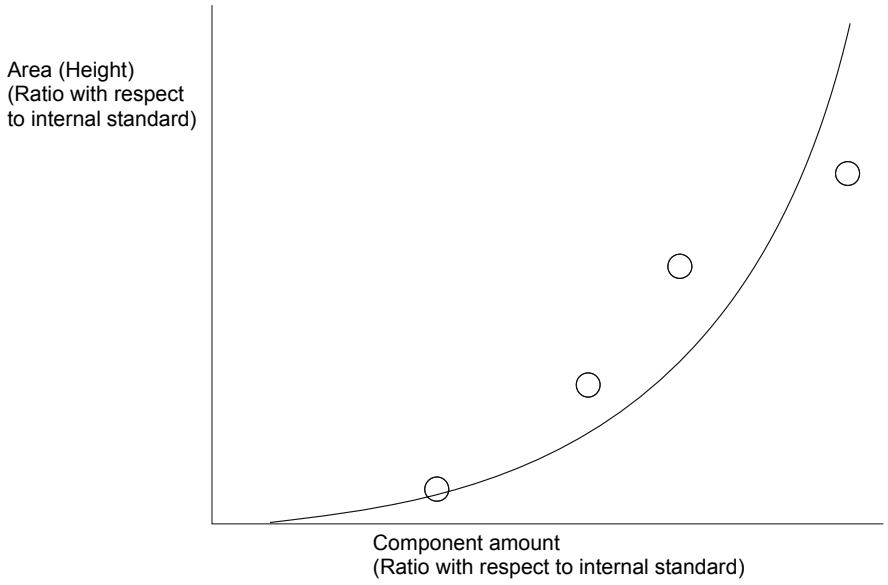


Figure A.19 Quadratic/Cubic Calibration Curve

Point-to-Point

Up to 64 different standard concentrations are analyzed, and a point-to-point calibration curve is created. No curve fit constant is displayed in the quantitation table when a point-to-point calibration curve is used, and none can be entered.

If there is only a single calibration level, a simple line that passes through that point and the origin is drawn.

If there are two or more calibration levels, a point-to-point curve is drawn; the initial line must pass through the origin.

Each point can represent the average of up to 10 individual analyses.

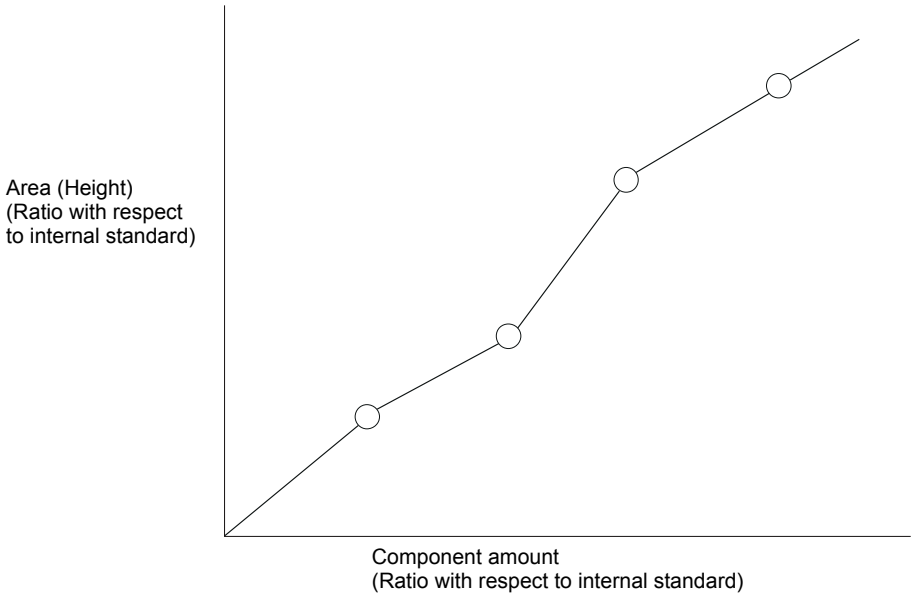


Figure A.20 Point-to-Point Calibration Curve

Note

Only with point-to-point calibration curves is the determined calibration curve constant not displayed in the calibration curve window. Further, the calibration curve constant cannot be entered.

Mean RF

Up to 64 standards with different concentrations are analyzed. First, linear curves are calculated passing through each individual point and the origin. Next, the simple average of the coefficients of the slopes of these lines is determined. The resulting calibration curve must pass through the origin.

If there is only a single calibration level, a simple curve that passes through that point and the origin is drawn.

Each point can represent the average of up to 10 individual analyses.

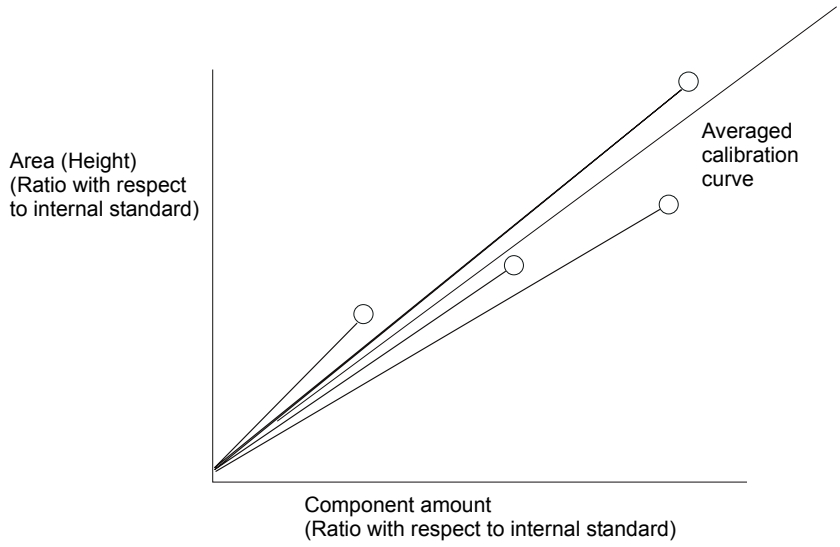


Figure A.21 Mean RF Calibration Curve

Appendix A Peak Processing and Mass Spectrum Operations

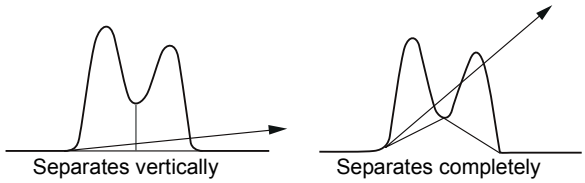
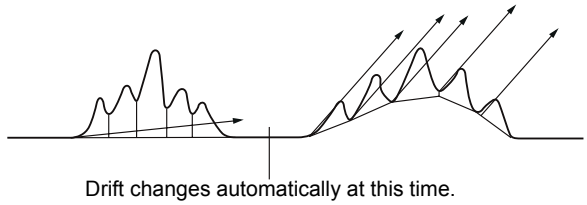
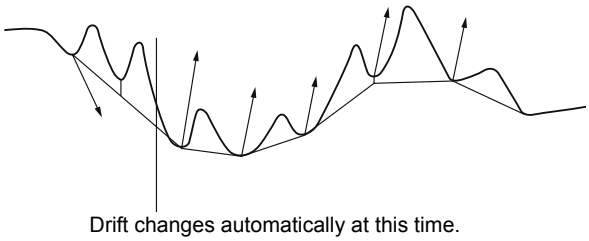
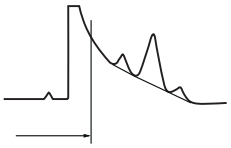
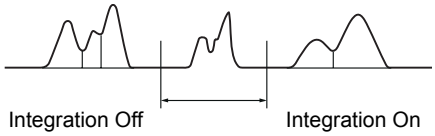
A.5 Troubleshooting

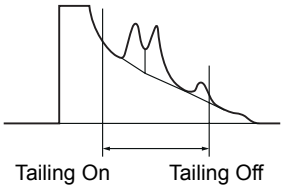
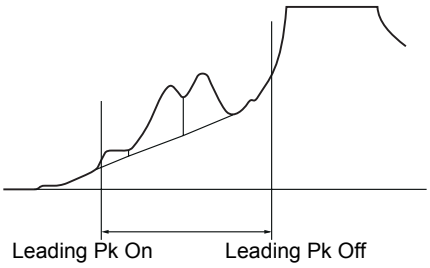
This section describes solutions for peak processing and peak identification problems.

A.5.1 Peak Processing

If peak processing is not performed properly with the method peak processing parameters, change the peak processing parameter settings according to this table. A peak time program may become necessary to change the peak processing parameters for certain components (for example, when the S/N ratio is different for each component).

#	Problem	Parameters	Solution
1	Narrowest peaks not detected.	Width Slope	Set the Width to the width at half-height of the narrowest peak. Decrease Slope by 1/2 in order to increase peak detection sensitivity.
2	Two or more peaks are detected as one peak.	Width Slope	Set the Width to the width at half-height of the narrowest peak. Decrease Slope by 1/2 in order to increase peak detection sensitivity.
3	Peaks that elute after the baseline starts drifting are not detected.	T.DBL	Set T.DBL to the time that it takes for peak widths to double. For temperature-programmed GC analyses, set T.DBL to a large value (near the end of data acquisition) to disable T.DBL.
4	Late-eluting broad peaks are not detected, or integration ends during peak elution.	Slope T.DBL	Decrease Slope by 1/2 to increase peak detection sensitivity. Set T.DBL to the time that it takes for peak widths to double, not to 0 (automatic).
5	Small peaks occurring after large peaks are not detected.	T.DBL	Set T.DBL to the time that it takes for peak widths to double, not to 0 (automatic). Use small peaks as the reference for selecting the T.DBL value.
6	Large baseline fluctuations detected as peaks.	Slope	Decrease detection sensitivity by gradually increasing the slope by 2x, 4x, . . . until fluctuations are not detected. Determine the slope value from the chromatogram according to the diagram below. 
7	Baseline is not calculated consistently or Baseline is unstable.	Drift	1) If the same peak in the same chromatogram is not processed reliably, disable automated Drift processing. See problem #8. 2) If reliable peak processing is not obtained for a peak that appears in several samples, or if the baseline needs to be calculated uniformly, see problem #8.

#	Problem	Parameters	Solution
8	The baseline needs to be calculated differently.	Drift	1) Set the Drift value as indicated below.  2) Use a time program to perform vertical separation or complete separation at different times.  
9	Certain peaks do not need to be integrated.	Suspending integration with Integration On/Off	1) To remove undesirable peaks that occur at the start of data acquisition (i.e., solvent peaks), use a time program to suspend integration from 0.1 min to a time slightly past the peak apex.  2) A time program is may also be used to suspend integration during an analysis. 

#	Problem	Parameters	Solution
10	Tailing processing is needed for a peak present on a tail (tailing processing is not performed automatically, or is not occurring correctly).	TAILING ON/ OFF/AUTO	Use a time program. Tailing is set so that the valleys and apexes of peaks on the tail are processed correctly as peaks on a tail.  **Note:** When performing tailing processing, the main peak must also be integrated. Do not disable peak processing with the LOCK command for the main peak.
11	Peaks should not be automatically processed as tailing.	Tailing	The same time program as above is used, and the program is divided into tailing ON, tailing OFF and tailing AUTO regions.
12	Leading (fronting) processing is needed for smaller peaks on a fronting peak.	Leading (fronting)	Use a time program. Set the Leading ON and Leading OFF parameters to include the apex and the valley that occur after the peaks on the fronting area. 

Note

Setting T.DBL when automatic processing is not used
T.DBL should be set to the time when the peak width has doubled in size relative to an original peak. If it is difficult to determine this time on the chromatogram, calculate the time as shown in the example below.

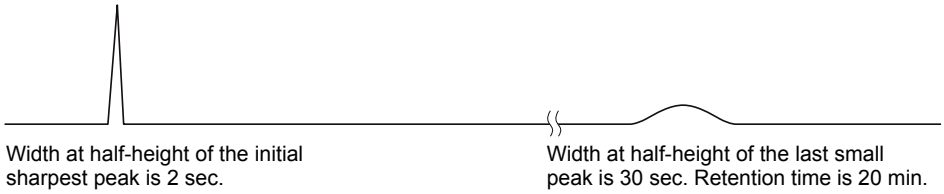


Figure A.22 Determining the T.DBL Value

(1) Use the width at half-height of the initial sharpest peak for the Width setting (normal setting). 2 sec is used in this example.
(2) Measure the retention time and width at half-height of the last small peak.

A retention time of 20 min and a half-width of 30 sec are used in this example.

Time required for 2x increase = 2 sec / 30 sec x 20 min x 2x = 2.7 min.

General formula:

$$\text{T.DBL} = \frac{\text{First Peak Width at Half-Height}}{\text{Last Peak Width at Half-Height}} \times \text{Last Peak Retention Time} \times 2$$

A.5.2 Peak Identification

If a target peak is not identified, or if a peak is identified incorrectly, make the following changes to the quantitative parameters, or change these settings in the Compound Table.

#	Problem	Parameter	Solution
1	Peak is not identified or is identified incorrectly.	Ret. Time	Ensure that the Compound Table contains correct parameters for the peak.
		Window	Is the retention time Window correct? Normally set to approx. 3 - 5 %.
		Band	Is the retention time Band correct? Normally set to the approximate peak half-width.
2	Wrong peaks are identified.	Min Area / Height	Increase minimum peak area/height to exclude small peaks.
		LOCK ON/ OFF	Suspend peak integration (lock ON/OFF) in a time program to exclude unwanted peaks.
		Window	Narrow the Window.
		Band	Narrow the Band.
3	Retention time fluctuates and does not allow proper identification.	Window	Widen the Window.
		Band	Widen the Band.
4	Retention time shifts cause the identification of one or more peaks, or the mistaken identification of a peak	Relative Retention Time Method	Use the relative retention time method for identification when the retention time shifts. Select a large peak that is separate from the other peaks as the reference peak. Alternatively, for complex chromatograms, the multi-reference relative retention time method uses up to eight reference peaks for proper identification.

Appendix B PDF Output

B.1 Setting up PDF Output

The GCMSsolution provides the capability to output a report in the PDF (Portable Document Format) when it is used with Adobe® Acrobat®, a tool for creation, management, and editing of PDF documents. When used with the CLASS-Agent, the GCMSsolution also provides the capability of outputting such reports to the printer and converting print images into a PDF file, which is then sent to the CLASS-Agent.

B.1.1 Preparation for PDF Output

The following products must be set up before the PDF output capability can be used.

• Adobe® Acrobat®(Version 5 or later)
To install this software, follow the instructions displayed on the screen that automatically appears when the CD-ROM for Acrobat® is placed into the drive, and perform the standard setup.

Note

This software package does not include Adobe® Acrobat®, which must be purchased separately. Adobe® Acrobat Reader™, which is included in the GCMSsolution installation disk, can only be used to read electronic documents of the PDF. It does not include the capability of creating a PDF file.

Note

To use the PDF output capability of this software, Acrobat® Distiller® is required. If Acrobat PDF Writer™ is used, PDF file output may not occur successfully.

• CLASS-Agent
To make archives in PDF format using CLASS-Agent, set it up in accordance with the installation manual for CLASS-Agent.

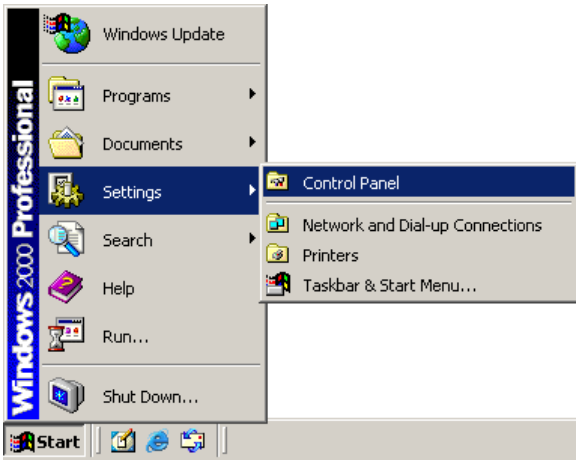
B.1.2 Install the PostScript Printer Driver

The figures in this chapter are displayed on Windows 2000. Operations are similar in Windows XP and Windows Vista.

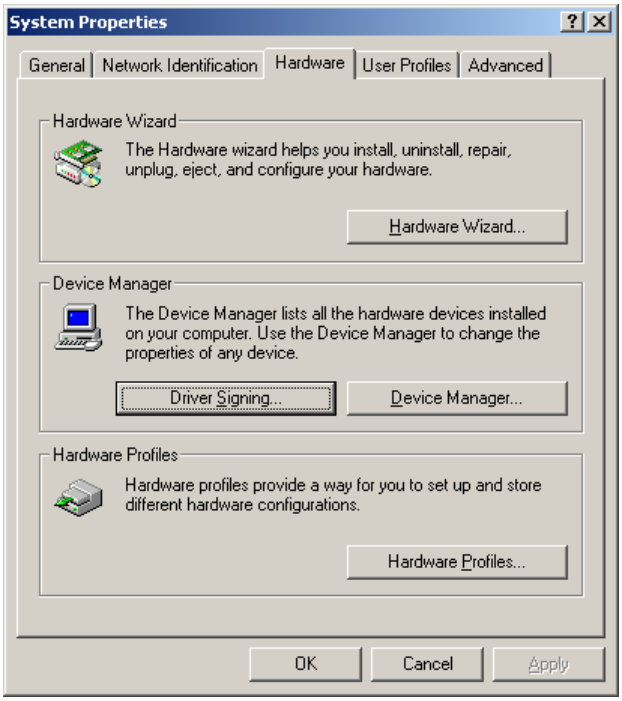
Preparations for installation

For Windows Vista, this installation preparation is not required.

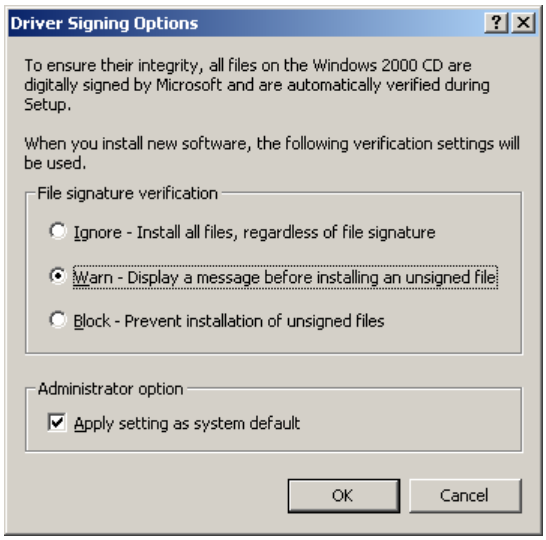
1. Select **Control Panel** from **Start - Settings** on Windows. Choose **System** from **Control Panel** to display "System Properties".
   In Windows XP, select **Control Panel** from **Start** on Windows. Choose **System** from **Performance and Maintenance** to display "System Properties."



2. Select the Hardware tab of the "System Properties" window and click the **Driver Signing** button.

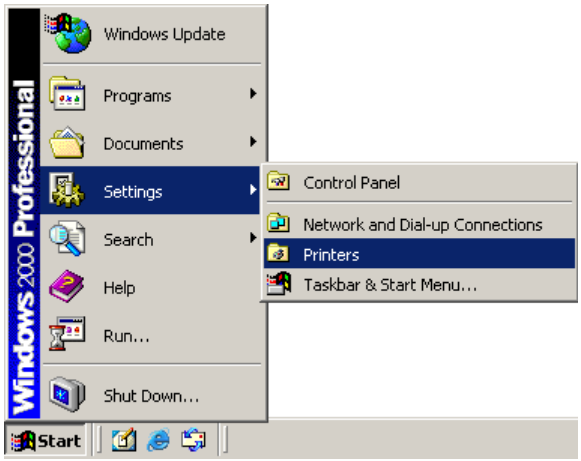


3. Check that "Block" is not selected in the File signature verification section of the "Driver Signing Options" window.

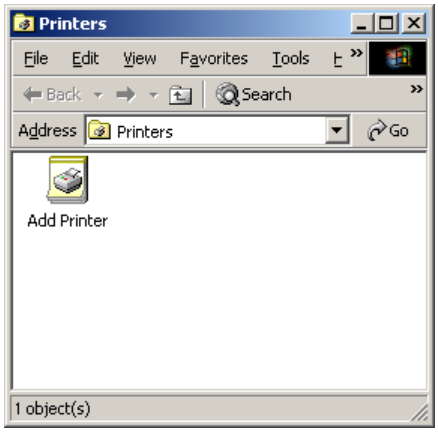


Installing the PostScript printer driver (for Adobe® Acrobat® 6.0)

1. Select **Printers** from **Start - Settings** in Windows.
For Windows XP, select **Printers and Faxes** from **Start** on Windows.
For Windows Vista, select **Start - Control Panel**. Switch **Control Panel** to **Classic View**, and select **Printers**.

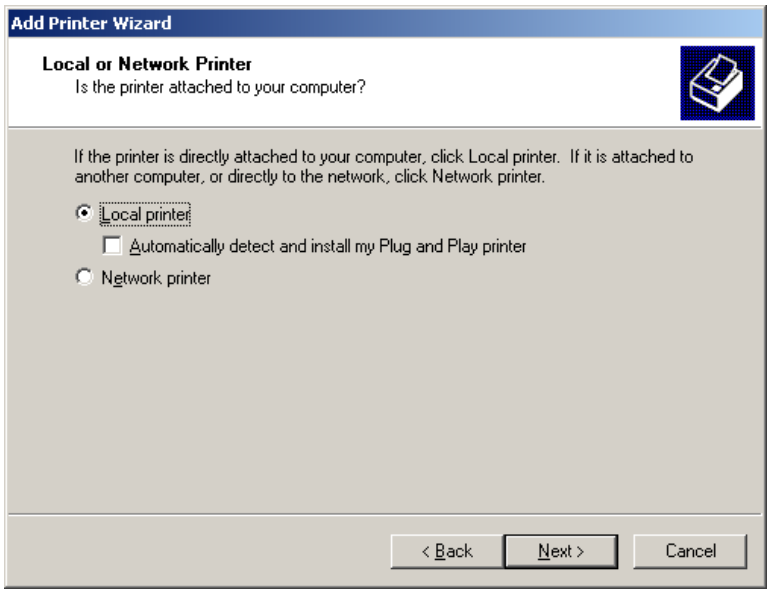


2. Double-click "Add Printer" on the "Printers" window to start "Add Printer Wizard". For Windows XP and Windows Vista, click "Add a printer" in **Printer Tasks** to start "Add Printer Wizard".

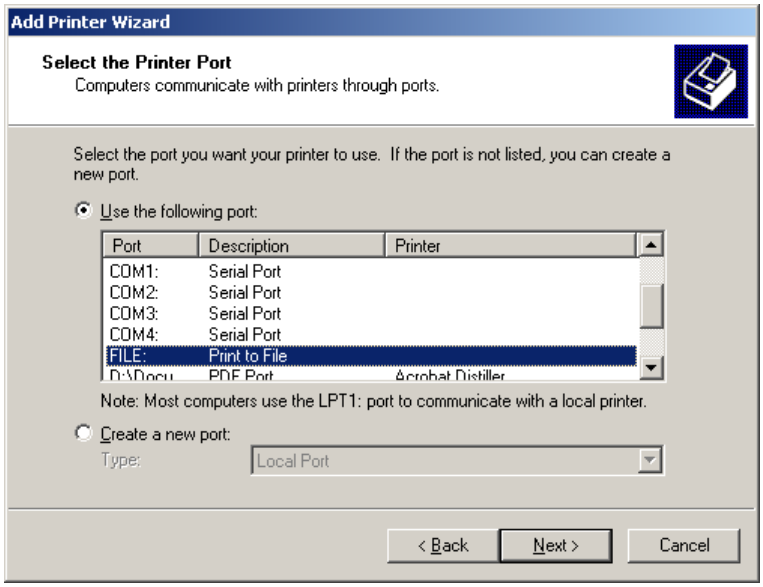


3. Click the **Next** button on the "Welcome to the Add Printer Wizard" window. For Windows Vista, this window is not displayed. Proceed to step 4.

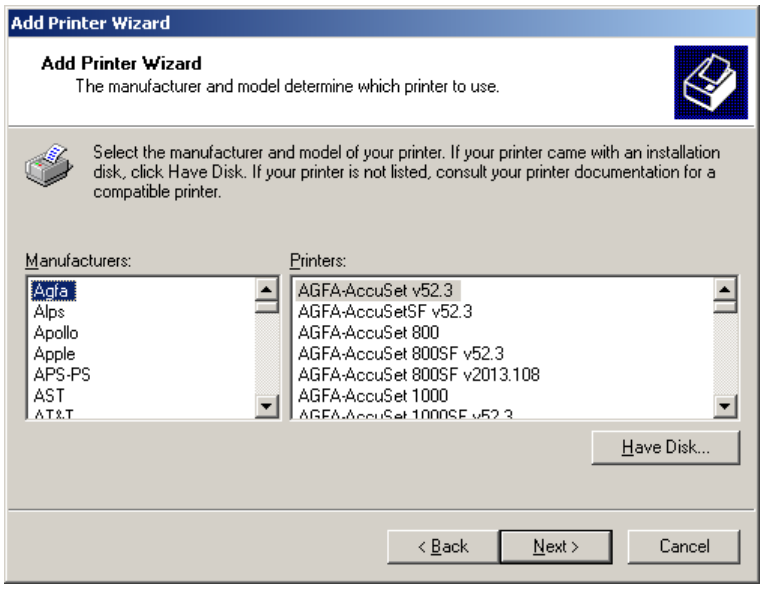
4. Select "Local Printer" on the "Local or Network Printer" window. Do not select the option, "Automatically detect and install my Plug and Play printer". Then click the **Next** button.



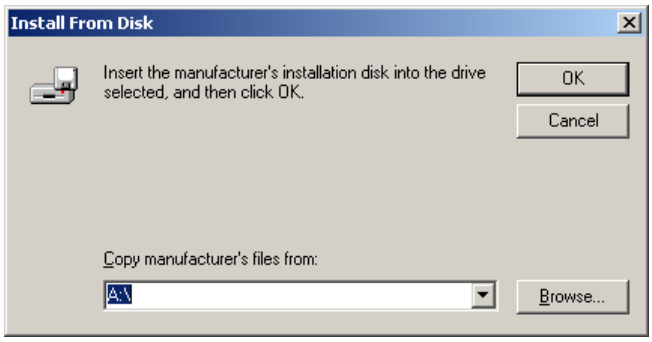
5. Select "Use the following port" on the "Select the Printer Port" window and specify "FILE: (Print to File)" for the Port. After the settings, click the **Next** button to proceed to "Add Printer Wizard".

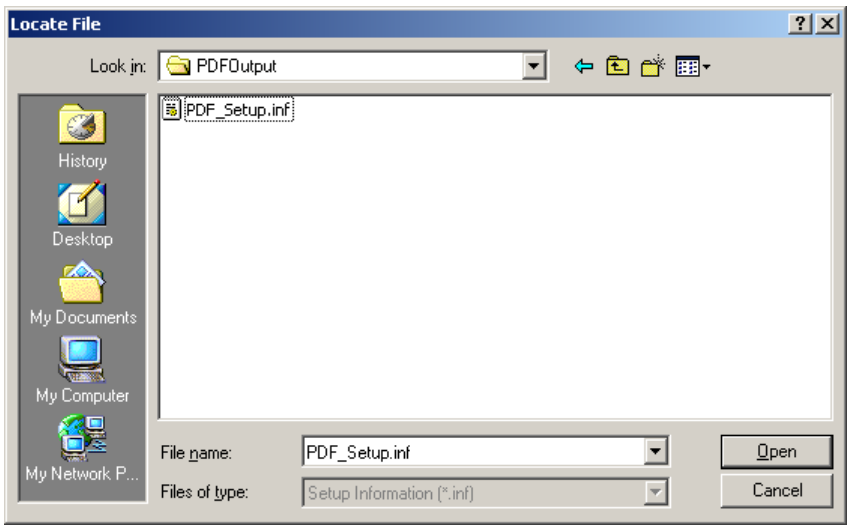


6. Click the **Have Disk** button on the "Add Printer Wizard" window to display the "Install From Disk" window.

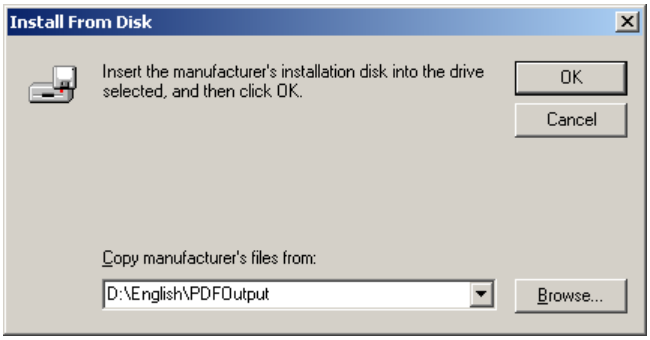


7. Click the **Browse** button on the "Install From Disk" window, select "PDF_Setup.inf" from the "\ENGLISH\PDFOutput" folder on the GCMSsolution installation disk, and then click the **Open** button.

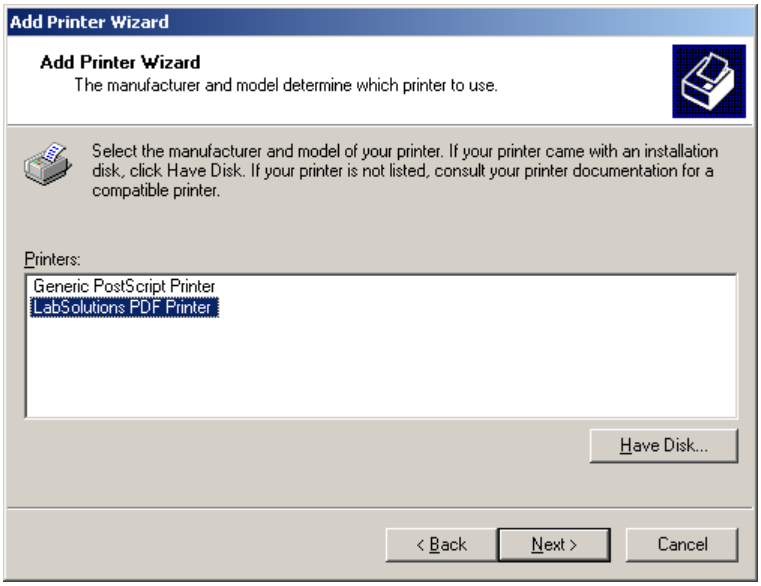




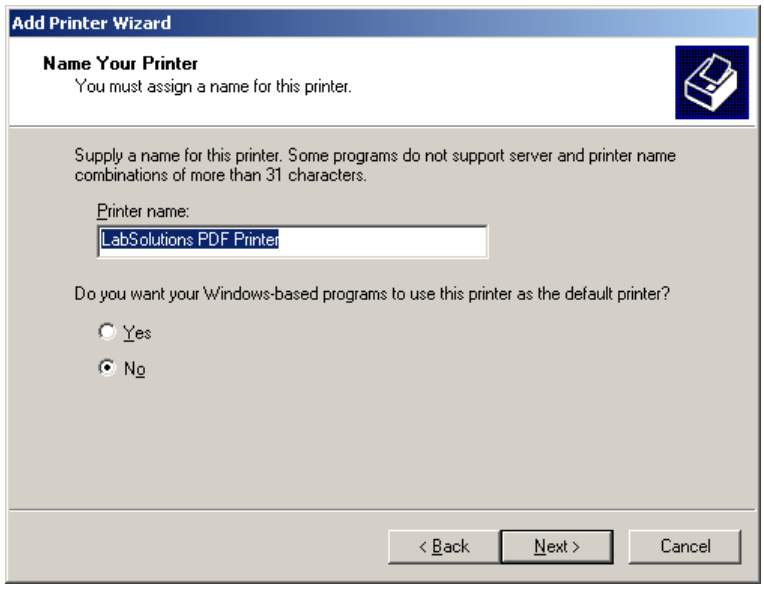
8. Check that the correct path to "PDF_Setup.inf" is selected on the "Install From Disk" window, and then click the **OK** button.



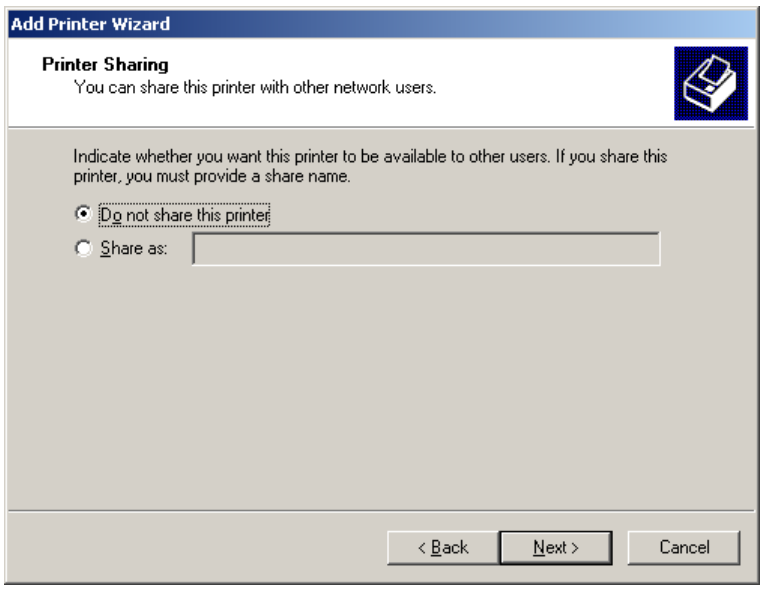
9. The "Add Printer Wizard" window lists the selectable printers. Select "LabSolutions PDF Printer" from the list and then click the **Next** button. If no appropriate printer is included in the list, still click the **Next** button.



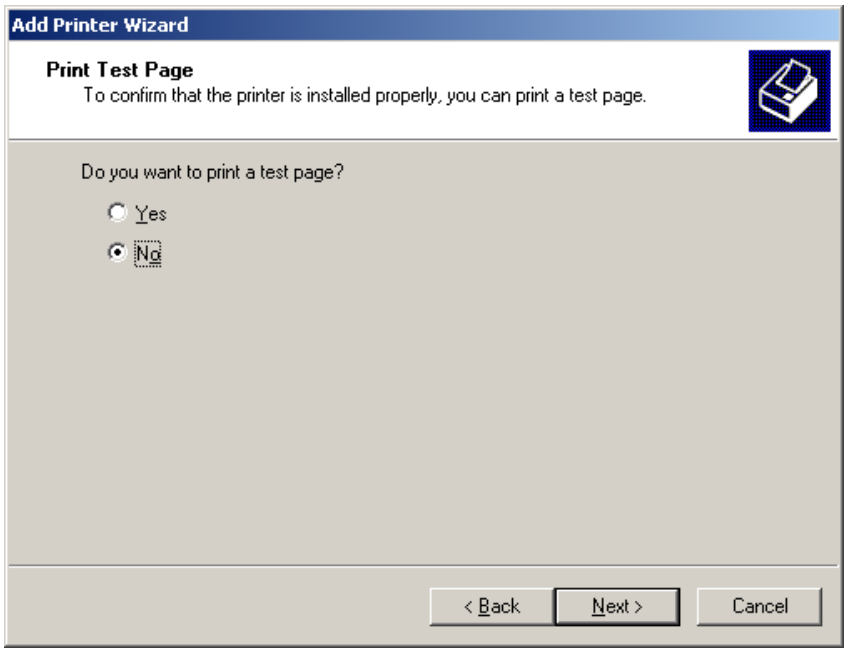
10. Check that the printer name selected in step 9 is correctly entered on the "Name Your Printer" window. This window allows you to edit the printer name if necessary. Select "No" for "Do you want your Windows-based programs to use this printer as the default printer?" Then click the **Next** button.
For Windows Vista, the window "Would you like to install this device software?" may be displayed after clicking the **Next** button. In this case, click the **Install** button.



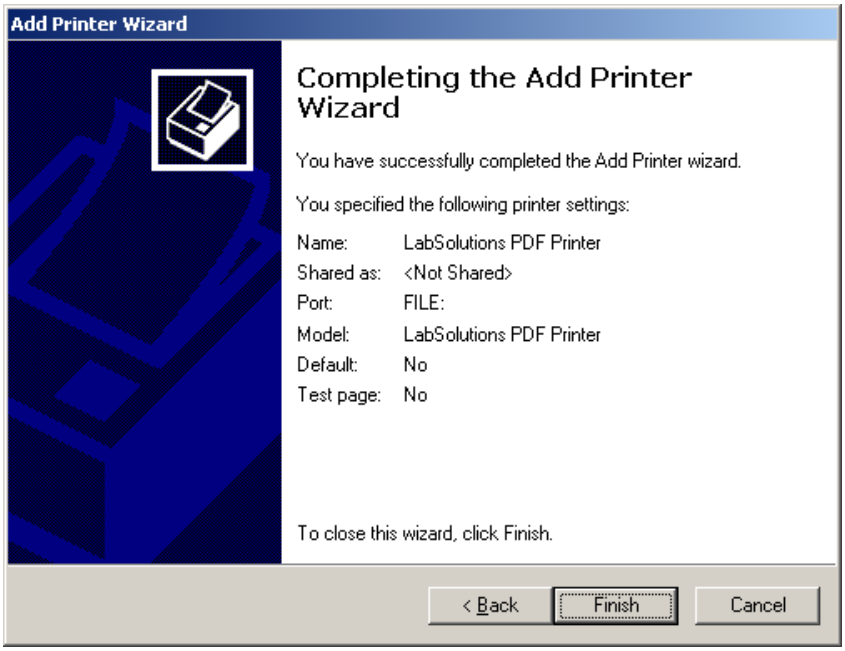
11. Select "Do not share this printer" on the "Printer Sharing" window and then click the **Next** button.
For Windows Vista, the [Add Printer] window is displayed. Click the **Finish** button, and proceed to step 15.



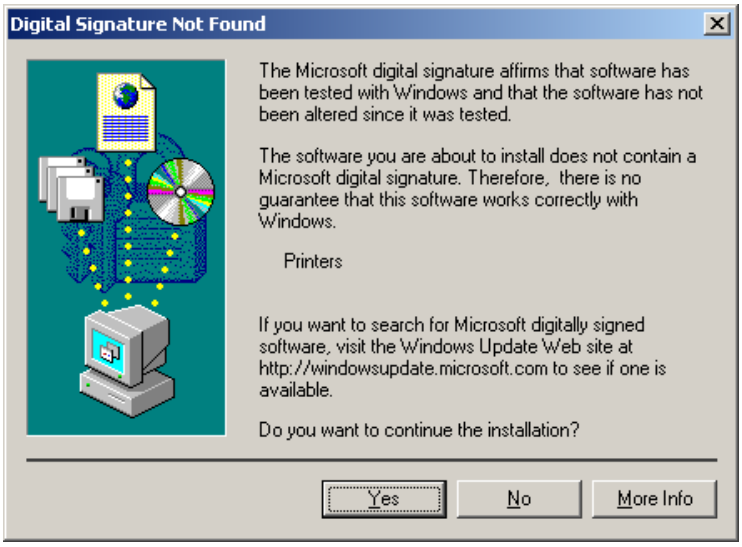
12. Select "No" on the "Print Test Page" window and then click the **Next** button.



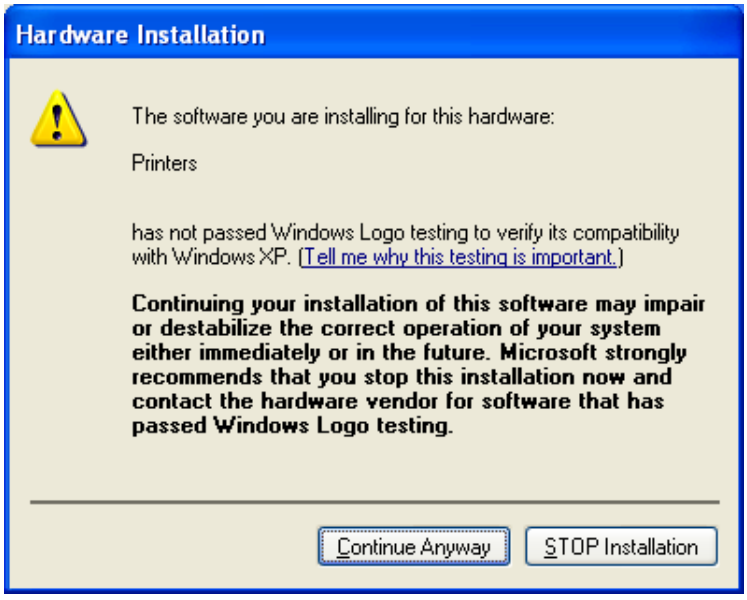
13. Click the **Finish** button to exit the wizard.



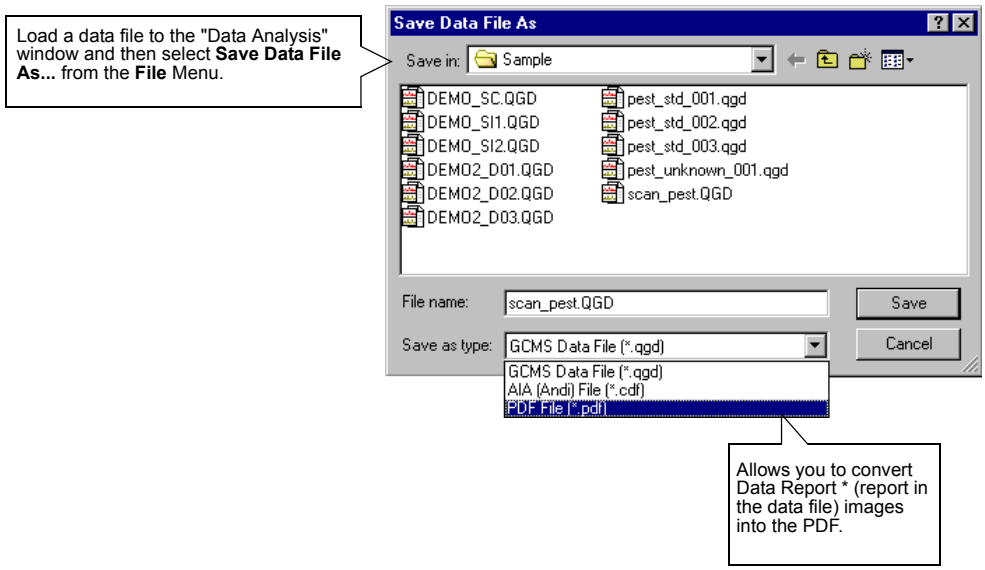
14. After you have clicked the **Finish** button, the following warning message may appear. In this case, click the **Yes** button. The installation will be completed successfully.



For Windows XP:
Click the **Continue Anyway** button.



15. After the installation has been completed, display the GCMS Postrun screen to see whether the PDF output settings are valid.

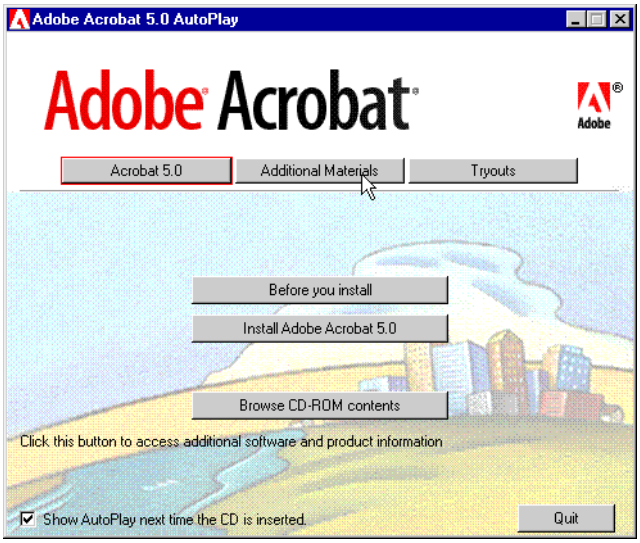


* If a data file in which no report format is recorded is converted into a PDF and then saved, a blank PDF file will be created.

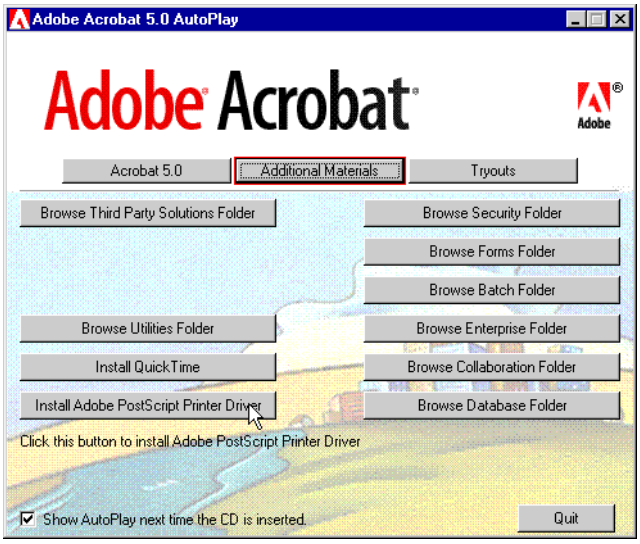
Installing the PostScript printer driver (for Adobe® Acrobat®5.0)

1. On the opening window that automatically appears when the CD-ROM for Acrobat® product is placed into the drive, click on the **Next** button to proceed to the next step.

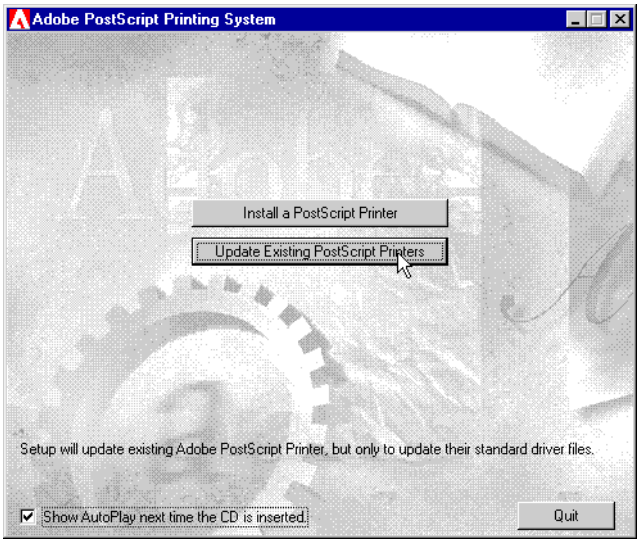
2. On the displayed window, select the **Additional Material** button. The list of buttons related to additional materials will appear.



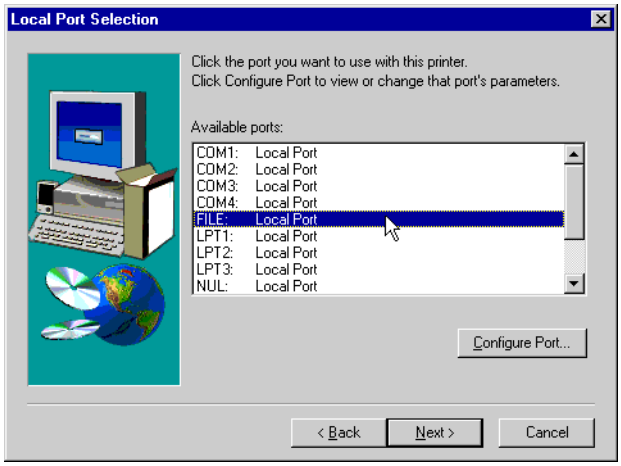
From the displayed buttons, select the **Install Adobe PostScript Driver** button and then specify a postscript printer for PDF output.



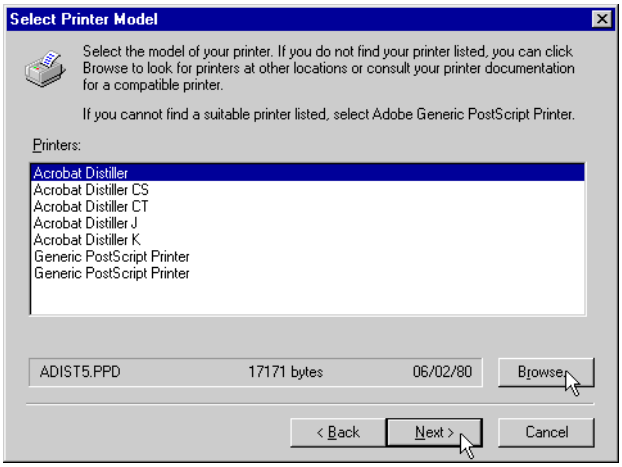
3. Click on the **Update Existing PostScript Printers** button. Follow the message on each window based on the default values until the "Local Port Selection" window appears.



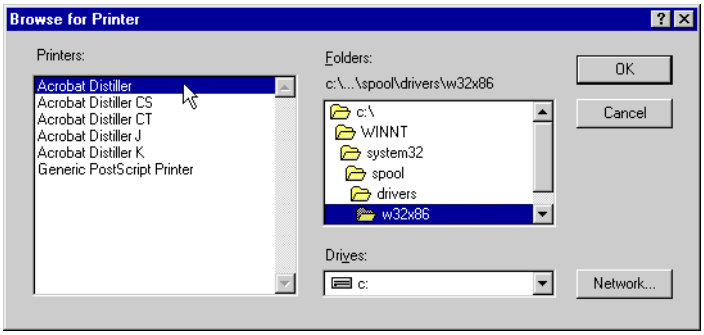
4. On the "Local Port Selection" window, select FILE: for the port. Click on the Next button. The "Select Printer Model" window will appear.



5. Click on the **Browse...** button on the "Select Printer Model" window to display the "Browse for Printer" window.

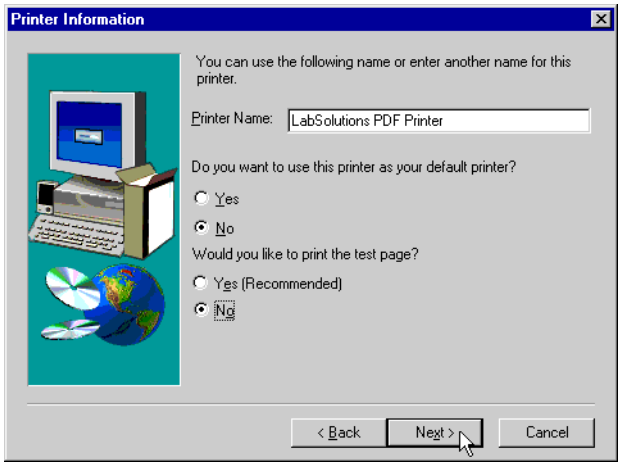


Change the drive/folder over to "C:\WINNT\system32\spool\drivers\w32x86". Now "Acrobat Distiller" is available for selection from the list of printers. Select it and then click on the **OK** button.

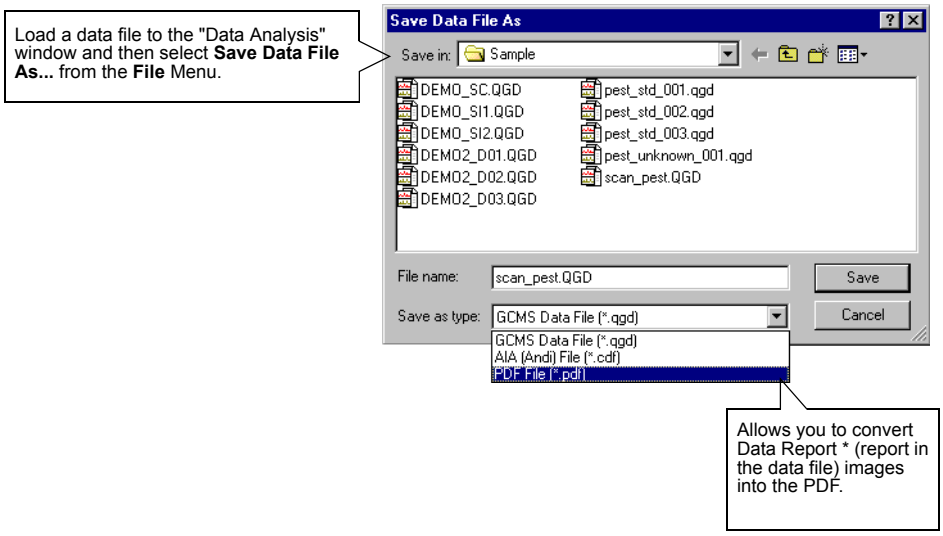


You will return to the "Select Printer Model" window. With "Acrobat Distiller" selected, click on the **Next** button and subsequently proceed using on the default values until the "Printer Information" window appears.

6. On the "Printer Information" window, specify a printer name. Enter "LabSolutions PDF Printer". Now the settings have been completed. Proceeding in accordance with the message that will appear on each window completes the installation of the printer driver. Each one blank space must be given before and after "PDF".



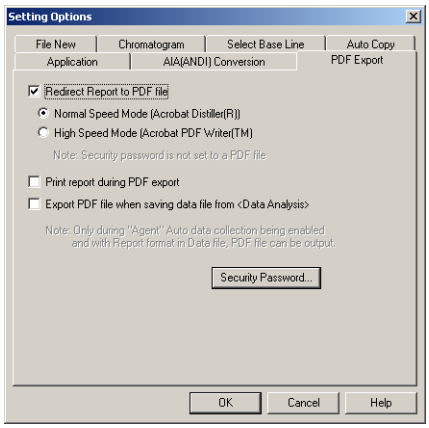
7. After the installation has been completed, display the GCMS Postrun screen to see whether the PDF output settings are valid.



If a data file in which no report format is recorded is converted into the PDF and then saved, a blank PDF file will be created.

B.1.3 [PDF Export] Tab

This tab is used to specify the PDF output settings.



1. Select the **Tools** > **Option** command.
2. Specify the parameters about PDF Output in the PDF Output tab.

Parameter		Description
Redirect Report to PDF file		When selected, the output format of reports is changed to PDF. **Note:** The following document information is included in the output file: **Title**: Filename of the original file **Subject**: Name of the application program from which the PDF file is output (Data Acquisition, Batch Analysis, Batch Postrun, or Report Generator) **Author**: Name of the user who was logged in when the PDF file is output **Keywords**: Status of data (postrun processing without file saving or presence/ absence of time program for integration in data) **Creator**: Product name (GCMSsolution) and version number Other information such as sample information is also included though they are not displayed.
	Normal Speed Mode (Acrobat Distiller®)	When selected, Acrobat® Distiller® is used to output PDF files. Note: When a password has been assigned to the Admin account, the "Changing the Document" security setting of the PDF file is set to "Not Allowed" by the Acrobat standard security feature using that password as the master password. Other security settings are applied in accordance with the settings specified in the Acrobat® Distiller®.

Parameter		Description
	High Speed Mode (Acrobat PDF Writer™)	When selected, Acrobat® PDFWriter™ is used to output PDF files. When Acrobat Ver.6 is used, "High Speed Mode" is not used. If this mode is selected, PDF file output may not occur successfully. **Note:** No security settings are applied to PDF files unlike the "Normal Speed Mode".
Print report during PDF export		When selected, a report is output to the printer at the time of PDF output, if the report output format is set to PDF file.
Export PDF file when saving data file from <Data Analysis>		If selected, a PDF file is output when a data file is saved in the "Data Analysis" window. **Note:** This setting is effective only when the saved data file contains a report format and the CLASS-Agent program is set to automatically collect data files.

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Appendix C Specifications

C.1 Performance

Standard model (Dual TMP model)

Mass Range	m/z 1.5 - 1024	
Resolution	R=2M (FWHM)	
EI scan sensitivity	S/N>60/1 (RMS) for 1pg octafluoronaphthalene molecular ion at m/z 272	
	Column	DB-5ms 30 m × 0.25 mm I.D., 0.25 µm
	Scan range	m/z 200 - 300
	Interval (Event Time)	0.5 sec
EI SIM sensitivity	S/N>60/1 (RMS) for 100fg octafluoronaphthalene molecular ion at m/z 272	
	Column	DB-5ms 30 m × 0.25 mm I.D., 0.25 µm
	Interval (Event Time)	0.2 sec
Maximum scan rate	10000 u/sec (single scan) Scanning max rate for scan range m/z 60 - 660 (600 u) at 0.1 sec interval	

Single TMP model

Mass Range	m/z 1.5 - 900	
Resolution	R=2M (FWHM)	
EI scan sensitivity	S/N>30/1 (RMS) for 1pg octafluoronaphthalene molecular ion at m/z 272	
	Column	DB-5ms 30 m × 0.25 mm I.D., 0.25 µm
	Scan range	m/z 200 - 300
	Interval (Event Time)	0.5 sec
EI SIM sensitivity	S/N>30/1 (RMS) for 100fg octafluoronaphthalene molecular ion at m/z 272	
	Column	DB-5ms 30 m × 0.25 mm I.D., 0.25 µm
	Interval (Event Time)	0.2 sec
Maximum scan rate	10000 u/sec (single scan) Scanning max rate for scan range m/z 60 - 660 (600 u) at 0.1 sec event time	

Appendix C Specifications

C.2 Hardware

Gas chromatograph	Model	GC-2010
	Oven temperature	Maximum 450 °C
	Linear temperature elevation rate (with power source voltage of 100, 115 V AC)	40 °C/min up to 200 °C 15 °C/min up to 350 °C 7 °C/min up to 450 °C
	Linear temperature elevation rate (with power source voltage of 230 V AC) (high power oven)	70 °C/min up to 200 °C 50 °C/min up to 300 °C 30 °C/min up to 400 °C
	Injection port temperature	Maximum 450 °C
GC/MS interface	Model	Capillary column direct interface
	Temperature	Room temperature - 350 °C
Ion source	Ionization type	EI
	Temperature	Temperature control: 100 - 260 °C
	Filament	Dual filament (automatic switching)
	Electron voltage setting range	10 - 200 V
	Electron current setting range	10 - 250 µA
Analysis part	Cylindrical quadrupole with pre-rod	
Detector	Secondary electron multiplier with conversion dynode	
Vacuum system	Main pump	Differential vacuum system 220 L/sec turbomolecular pump 65 L/sec turbomolecular pump For Single TMP models, 65 L/sec turbomolecular pump only.
	Backing pump	30 L/min (60 Hz) oil rotary pump

Appendix C Specifications

C.3 Workstation

C.3.1 PC

PC	CPU	Pentium III (600 MHz) or better
	Internal memory	256 MB or greater
	Hard drive	3 GB or greater
	Operating System	Windows 2000 SP1 or later
		Internet Explorer 3.02 or later
Monitor	15 inch LCD or 17 inch CRT, resolution 1024 × 768 or greater	
Printer	Laser printer, A4	
*PC may be upgraded without prior notice.		

C.3.2 Software

Analysis	Analysis Conditions	GC measurement conditions, MS measurement and data processing parameters are all saved together in a method file.
	MS measurement mode	Scan, SIM (64 channel × 128 groups)
	GC detector	FID, FPD, FTD, ECD, TCD
	Continuous analysis	Continuous automated runs using batch processing
Instrument control	Start/stop vacuum system	
	GCMS system automatic and manual tuning	
	GCMS system check	
Data Processing	Data analysis	Chromatogram/mass spectrum graphing
		Chromatogram/mass spectrum comparison
		Mass spectrum background deletion and averaging
		Chromatogram area calculation
		Column performance calculation
	Library search	Search modes: similarity index, index search
		Up to 5 libraries can be search simultaneously
		Structural formula display
		Private library editing

Data Processing	Quantitation	Maximum number of peaks identified: 1000
		Identification method: absolute retention time/ relative retention time method, time band/time window methods, reference ions (5 max), mass pattern matching with registered spectra
		Quantitation calculations: corrected area normalization with scale factor, internal standard, absolute calibration, standard addition, area percent
		Calibration curves: linear (least squares method, average coefficient method), point to point, quadratic, cubic Weighted (1/C, $1/C^2$, 1/A, $1/A^2$)
		Calibration points: 64 maximum (averaging of up to 10 measurements for each point)
		Calibration curve correction
		Grouping processing
	Report generation	Chromatograms, mass spectra, peak report, similarity search results, quantitation calculation results, calibration curves, etc.
		Format editing
	Batch processing	Data processing, report generation continuous automated processing
	Data conversion	ANDI (AIA) conversion, quantitation result ASCII conversion, PDF output of report image
System	Data Explorer	File backup and deletion of various file types
	QA/QC	Precision management, quality management, recovery rate, spectrum check, etc.
	Security	Specify users by user name and password, and determine levels of authorization.
	Audit trail	Save the changes (and reasons for change, if desired) each time a method file is saved.

C.4 Installation Requirements

Power supply	Frequency	50/60 Hz
	For GC	Single phase AC 100, 115 V 1800 VA
		Single phase AC 230 V 2600 VA (for high-powered oven)
	For MS	Single phase AC 100 to 240 V 1000 VA
	Voltage fluctuation	Operating power voltage range ±10 %
		Specification power voltage range ±5 %
	* A dedicated power source with a circuit breaker should be provided for the unit. Do not share power supply with another device.	
	* With regards to voltage fluctuations, the device is designed to work in a range ±10 % including fluctuations caused by high frequency noise, but it is recommended that it be kept within ±5 % including noise to ensure the best efficiency. The current frequency fluctuation should be kept within ±0.5 Hz.	
	* Power supplies for PC, monitor and printer are also required.	
	Grounding	100 Ω or less
Environmental requirements	Temperature	Constant within 18 to 28 °C
	Humidity	40 to 70 %RH (No condensation)
	* An exhaust duct must be installed. The pump exhaust must be connected to the exhaust duct to avoid oil mists and injected solvents and samples from being directly vented into the room. If this is not possible, the oil mist filter below should be installed. Injected solvents and samples are vented directly into the room with the oil mist filter. (P/N 042-00124-31, Oil mist filter EMF3)	
	* Dust, vibrations, spatial noise and corrosive gases should be also avoided.	

C.4.1 Installation Examples

Installation Example (dimension will vary depending on the PC and printer used.)

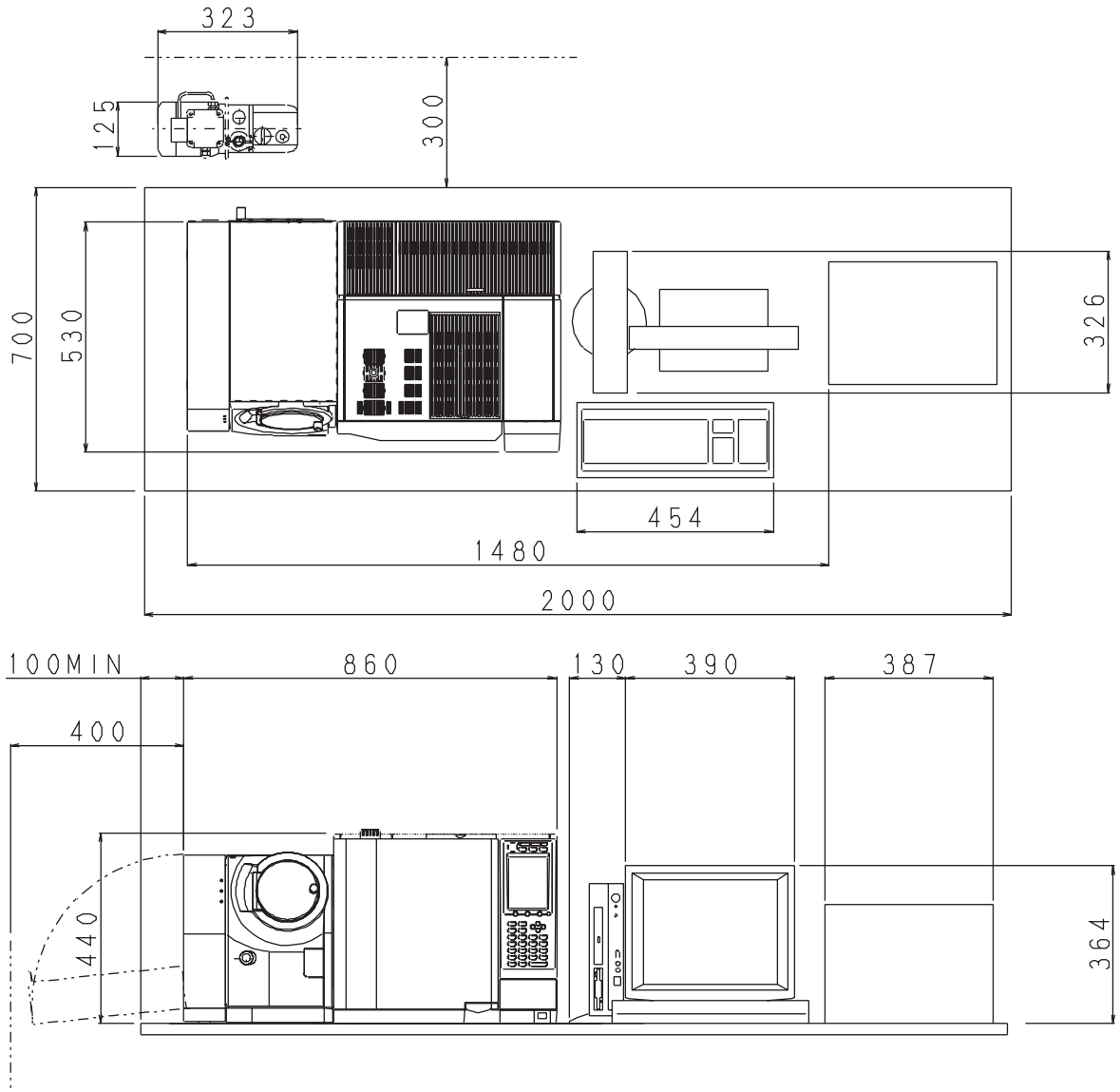

Figure C.1 Installation Example

Tubing and cable lengths are as follows.

MS rotary pump	Vacuum tubing	1.5 m approx.
	Exhaust tubing	4 m approx.
	Power cord	1.5 m approx.
PC-MS	Cable	2 m approx.

Appendix D Consumable Parts and Maintenance Parts List

D.1 Consumable Parts List

This section presents the consumable parts list and maintenance parts list for a standard GCMS-QP2010 configuration. Parts for optional units are found in their accompanying user manuals. Most of the consumable and maintenance parts for the GC component (GC-2010) are shown in the list, but for additional details, refer to the GC-2010 User Manual included separately (221-40406). If there are questions concerning parts on this list, please ask your Shimadzu representative.

Unit Name	Part No.	Part Name	Remarks
GC	221-35507-02	SEPTUM SET, (LB-2), 50/PKT	
	036-11203-84	O-RING, 4D P5, 5/PKT	For glass insert attachment
	221-41444-01	GLASS INSERT, SPL-2010	For Split injection
	221-48335-01	GLASS INSERT, SLESS/WBI	For Splitless injection
	221-48600	DEACTIVATED SILICA WOOL, 2G	
	221-48876-03	DEACTIVATED GLASS INSERT, SLESS	5/PKT
	221-49065-91	AU GASKET 5/PKT	1PC 221-48990
	221-34121-93	MOLECULAR SIEVE FILTER, /SPL-14	For removing carrier gas impurities
	221-42559-92	FILTER ASSY (SPLIT)	Split flow path
	221-42559-92	FILTER ASSY (PURGE)	Septum purge flow path
	201-35183	GASKET, AL, COLUMN PACKING, 100PC	For line connections
	221-48974	SPACER 1.5	Spacer under Septum
DIRECT INTERFACE	670-15003-03	FERRULE, GVF/004, 10/PKT	For less than 0.25 mm bore capillary column
	670-15003-04	FERRULE, GVF/005, 10/PKT	For 0.32 mm bore capillary column *1
	670-15003-07	FERRULE, GVF/008, 10/PKT	For 0.53 mm bore capillary column *1
	670-11009	NUT, SSNE16/012, 5 PCS	
ION SOURCE	225-10340-91	FILAMENT	
	225-10445	BOX	
	225-10442-91	REPELLER ASSY	
	225-10255	REPELLER	REPELLER only
	225-10446-91	BOX (HEAT TREATED)	
	225-10447-91	REPRLLER (HEAT TREATED)	
	225-01068	INSULATOR	
CALIBRATION GAS	225-09493-03	STANDARD SAMPLE, PFTBA (5 g)	
	225-09493-01	STANDARD SAMPLE, TRIS (0.5 g)	
VACUUM	017-30163-11	ROTARY PUMP OIL ULTRAGRADE15 1 L	
JIG, TOOLS	085-35124-03	ABRASIVE CLOTH, ULTRA FINE, 20/PKT	
	225-10194-91	JIG, IS	
	225-11657-08	JIG FOR COLUMN CONNECTION (FOR I/F)	
	225-11657-09	JIG FOR COLUMN CONNECTION (FOR INJ)	

*1: For Dual TMP model only

D.2 Maintenance Parts List

Unit Name	Part No.	Part Name	Remarks
GC	221-46260-92	PCB KEY ASSY, GC2010	
	078-12146-01	LCD, LM32019P	
	221-46470-02	KEY RUBBER 1, GC2010	
	221-46471-02	KEY RUBBER 2, GC2010	
	221-43695-91	PT SENSOR ASSY, 17A+	
	221-43696-91	THERMO COUPLE WITH CONTACT, V2	
DIRECT INTERFACE	225-10547	INSULATOR, I/F	
	018-23651	ADHESIVE TAPE	
	036-11251	O-RING, 4D P50	
	225-10549-91	HEATER BLOCK ASSY	For 100 V – 115 V (includes heater and Pt sensor)
	225-10549-92	HEATER BLOCK ASSY	For 220 V – 240 V (includes heater and Pt sensor)
ION SOURCE	200-44394	CERAMIC, INSULATOR, A24-251-1	
	225-10439-91	HEATER BLOCK ASSY	
	225-10436-91	PT ASSY, IS	
	018-17301	FOIL, AL 250X10MT	
	225-10200-91	IS ASSY	Lenses, Magnets and Heater unit
	225-10554-91	CUP, I/F A ASSY	
	034-01602-31	SPRING, SUS UR8-10	
	225-10434-91	CABLE ASSY, F1(S)	
	225-10434-92	CABLE ASSY, F2(S)	
	225-10434-93	CABLE ASSY, F1(L)	
	225-10434-94	CABLE ASSY, F2(L)	
	225-10434-95	CABLE ASSY, L1	

Unit Name	Part No.	Part Name	Remarks
DETECTOR	225-10463-91	EM (H) ASSY	Detector as a whole
	225-09340-11	EM, AF620	Detector; Secondary electron multiplier only
	225-10466-91	SIG CABLE ASSY, DET	
	225-10466-92	LENS CABLE ASSY, DET	
	225-10466-93	HV CABLE ASSY, DET	
	225-10466-94	CDD CABLE ASSY, DET	
	036-11271	O-RING, 4D P105	
	225-10464-91	FEEDTHROUGH ASSY, CDD	
	225-09148-92	FEEDTHROUGH	
	036-11218	O-RING, 4D P18	
MS FILTER	225-01132	PIN/QP-5000	
	225-10407-91	CABLE ASSY, MS FILTER	
	225-10408-91	SHORT CABLE1, PRE-ROD	
	225-10408-92	SHORT CABLE2, PRE-ROD	
	036-11266	O-RING, 4D P90	
	225-09148-92	FEEDTHROUGH	
	036-11218	O-RING, 4D P18	For feed-through
	225-01373	SHORT SPRING	
	225-10390-01	TERMINAL PLATE (PRE)	
	225-11659	PRE ROD	
	225-10401	INSULATOR	For Pre Rod
	225-10402	SCREW(PRE)	For Pre Rod
	225-10403	COLLAR	For Pre Rod
CALIBRATION GAS SYSTEM	225-10179-91	VALVE ASSY, SI	
	225-01559-91	CAPILLARY ASSY	
	035-62971-05	SLEEVE SET, T-100SET	
	225-04257-91	GLASS BOTTLE, 5 PCS	
	036-11203	O-RING, 4D P5	

Unit Name	Part No.	Part Name	Remarks
VACUUM SYSTEM	225-09490-01	ION GAUGE	*1
	200-47686-02	PB-1 PIRANI TUBE FILAMENT	
	225-09509-02	TURBOVAC TW70H	TMP MAIN BODY
	225-09508-03	TURBO DRIVE 300	TMP CONTROLLER
	225-09587-01	TMP, TMH262	TMP MAIN BODY and TMP CONTOROLLER *1
	225-09587-04	LUBRICANT RESERVER	For TMH262 *1
	225-09517-01	RP E2M1.5 (100-120)	WITHOUT CABLE
	225-09517-02	RP E2M1.5 (220-240)	WITHOUT CABLE
	225-11446-91	CABLE, MS-RP	CABLE FOR RP
	221-09895-09	LABEL, AC220 240V	LABEL FOR CABLE
	221-09895-10	LABEL, AC100 115V	LABEL FOR CABLE
	210-13532-71	LABEL, RP1	LABEL FOR CABLE
	204-30020	HOSE NIPPLE, 16KF-18	for RP
	035-06004-51	CENTERING RING, KF10/16SNRCR	for RP
	035-02415-01	CLAMP RING, 10/16KF	
	016-31697-03	HOSE, PVC CHEMIFLEX 19X26	
	037-61024	HOSE CRAMP, HB-1-28	
	017-30290-11	LUBRICATING OIL BARRIERTA IS/V	
	225-03538-91	ELBOW, KF16-#15	*1
	225-03535-91	ELBOW, KF25-#15	
	225-11676	STRAIGHT JOINT, KF16-#15	*2
	035-06004-22	CENTERING RING, KF16SVCR	
	035-06004-24	CENTERING RING, KF25SVCR	
	035-02415-02	CLAMP RING, 20/25KF 18342	
	035-02411-21	CENTERING RING, 32036-PAZV	
	035-02411-22	CENTERING RING, 32040-PAZV	
	036-11271	O-RING, 4D P105	
	225-10125	FEEDTHROUGH 12P	
	036-11243	O-RING, 4D P40	
	225-10116-91	LEAK VALVE ASSY	
	036-11203	O-RING, 4D P5	

Unit Name	Part No.	Part Name	Remarks
MAIN POWER	225-11260-91	MAIN POWER ASSY	TW300/TW70H *3
	225-11260-92	MAIN POWER ASSY S	*2
	225-11260-93	MAIN POWER ASSY	TW300/TW70H *4
	225-11260-94	MAIN POWER ASSY	TMH262/TW70H *1
	074-80422-01	POWER SUPPLY, LEA50F-5	
	074-80422-51	POWER SUPPLY, LEA50F-24	
	074-80424-01	POWER SUPPLY, LEA150F-24	
	225-11360-91	PCB ASSY, LED-A	
	225-11350-91	PCB ASSY, PUMP CTRL-A	
	225-10333-91	FAN ASSY POWER	
HV PCB	225-11195-91	HV PCB ASSY	
RF PS	225-11075-92	RF PS ASSY	
	225-11060-91	PCB ASSY, RF DIFF-A	
	225-10296-91	FAN ASSY	
MAIN CTRL	225-11130-91	PCB ASSY, MAIN CTRL-A	
CPU PCB	225-11000-91	PCB ASSY, CPU-A	
IS CTRL	225-11225-92	IS CTRL-A ASSY	
DC POWER	225-11250-91	DC POWER-A	
IG CTRL	225-11280-91	PCB ASSY, IG CTRL-A	*1
PRE AMP	225-11030-91	PCB ASSY, PRE AMP-A	
CABLE	071-60814-05	CORD, KP-4819D+KS-31A	For 220 V – 240 V
	071-60816-12	CORDSET, UC-975-N01	For 100 V – 115 V
	225-19050	IEEE1394 Cable	
	088-52848-21	IEEE1394 Cable (4.5 m)	
	225-19051	RS232C Cable	

*1: For Dual TMP model only

*2: For Single TMP model only

*3: For 225-10000-xx, 225-10001-xx, 225-10002-xx
225-10040-xx, 225-10041-xx, 225-10042-xx
xx=24, 34, 37, 39, 91, 92

*4: For 225-10005-xx, 225-10006-xx, 225-10007-xx
225-10085-xx, 225-10086-xx, 225-10087-xx
xx=24, 34, 37, 39, 91, 92

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E.1 Vacuum Characteristics

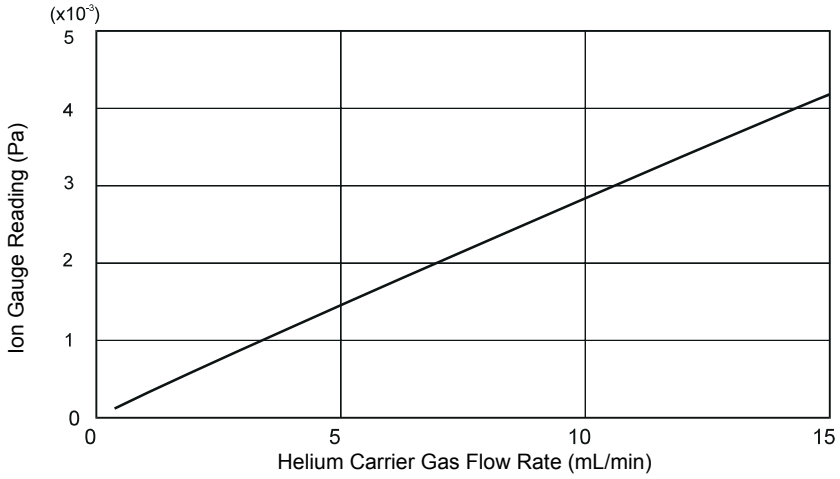
The graph below shows the vacuum dependence on carrier gas flow rate. A variation of roughly ±20 % in the vacuum may be observed depending on the ionization interval.

Outgassing from vacuum components may affect the vacuum, especially at low flow rates or when monitored in a short time after starting evacuation.

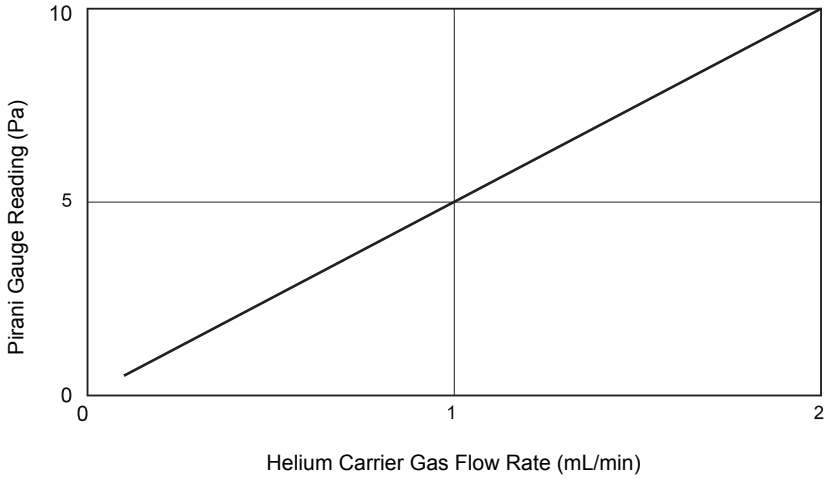
The maximum column flow rate at which data acquisition is possible is 15 mL/min for Dual TMP model. If the internal diameter of the column is 0.53 mm, use a column length greater than 25 m. In case of Single TMP model, it is 2 mL/min.

Carrier Gas Flow Rate and Degree of Vacuum

(Dual TMP model)

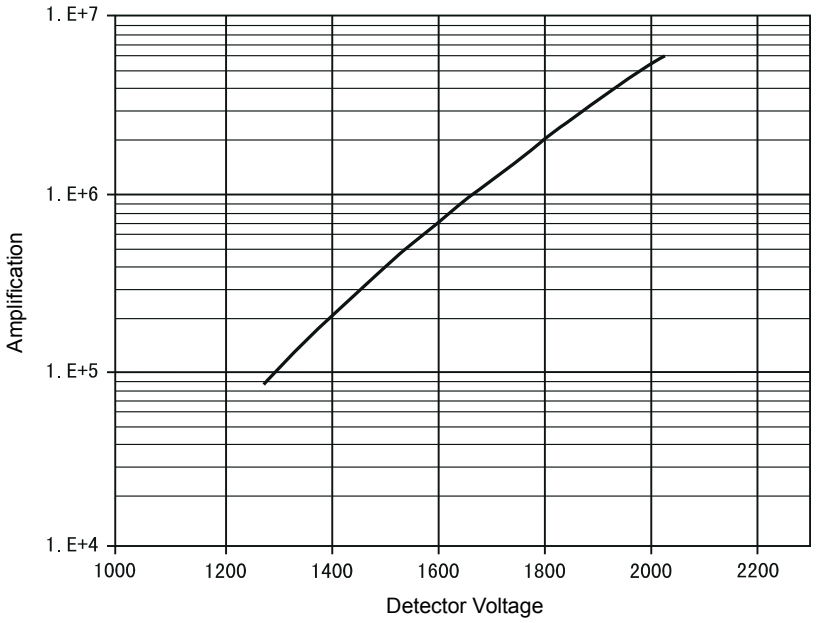


(Single TMP model)



E.2 Detector Amplification

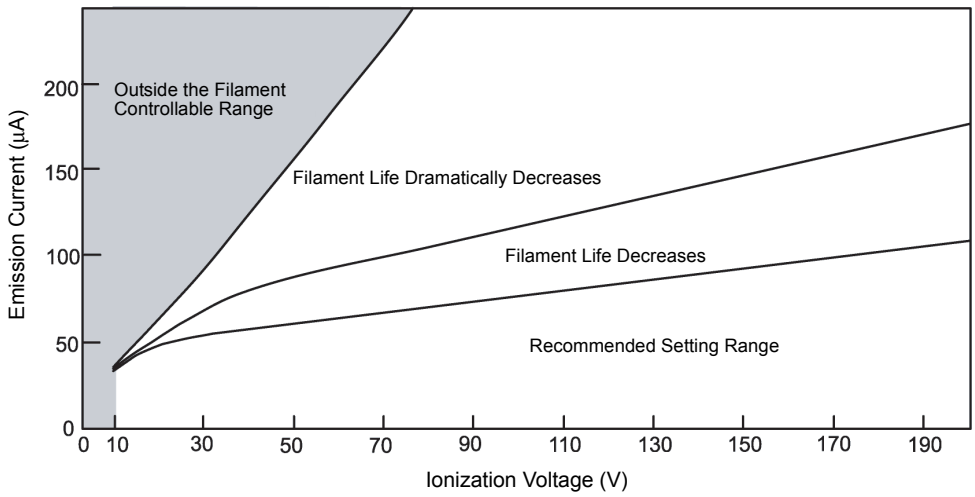
The characteristic graph shown in the figure below indicates average values. There will be slight variations depending on the detector.



E.3 Ionization Voltage/Emission Current and Filament Life

Ionization voltage and emission current can be changed; however, parameter setting may affect the life of the filament. Refer to the graph below when changing these parameters.

Ionization Voltage/Emission Current and Filament Life



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Appendix F Glossary of GCMS Terms

F.1 Abbreviations

AMU	Atomic Mass Unit
AOC	Automatic Operation Controller
CPU	Central Processing Unit
CRT	Cathode-Ray Tube
EI	Electron Impact Ionization
eV	Electron Volts
GC	Gas Chromatograph
GC/MS	Gas Chromatograph / Mass Spectrometer
I/F	Interface
IS, ISTD	Internal Standard
MC	Mass Chromatogram, i.e. the plot resulting from MS analysis
MS	Mass Spectrometer, i.e. the instrument
m/z	Mass-to-charge Ratio
NIST	National Institute of Standards and Technology
OS	Operating System
PC	Personal Computer
PFTBA	Perfluorotributylamine
RF	Radio Frequency
RP	Rotary Pump
SIM	Selected Ion Monitoring
S/N	Signal-to-Noise ratio
SP/SPL	Split / Splitless
TIC	Total Ion Chromatogram
TMP	Turbomolecular Pump

F.2 Glossary

A

Absolute retention time method

This is a method of identifying peaks. A peak is identified if it falls within an allowable retention time band or window defined on the basis of a preset expected retention time. This widely used method does not require reference peaks. A peak is identified using the absolute retention time method when it fulfills the relationship shown below.

$$\left(\begin{array}{c}\text{Expected retention}\\ \text{time of target peak}\end{array}\right) - \left(\begin{array}{c}\text{Actual retention}\\ \text{time of target peak}\end{array}\right) < \left(\begin{array}{c}\text{Allowable retention time}\\ \text{window/band of target peak}\end{array}\right)$$

See also: Time Window method, Time Band method

AMU (atomic mass unit)

This is the unit in which atomic masses are measured, defined as 1/12 the mass of Carbon 12. The mass of one Carbon 12 atom is exactly 12 u. The atomic mass of hydrogen is 1.007825.

AOC

Refers to one of the autosamplers that work with the GC system. The AOC injects samples sequentially into the GC injection port. Injections are reliable and automated.

Area Normalization

See Normalization method.

Autotuning

The Autotune program optimizes the performance of the ion lenses. PFTBA calibration gas is introduced and the ion source lens voltages, RF parameters and analyzer voltages are automatically adjusted. The user can edit tuning conditions for resolution adjustment, mass calibration, and sensitivity adjustment (target mass or mass pattern). The final tuning results are printed and can be saved as a tuning file.

Average RF method

This is a calibration curve fit type for quantitation. Up to 64 standards with different concentrations are analyzed. First, linear curves are calculated passing through each individual point and the origin. Next, the simple average of the slopes of these lines is determined. The resulting calibration curve must pass through the origin. If there is only a single calibration level, a simple curve that passes through that point and the origin is drawn. Each point can represent the average of up to 10 individual analyses.

B

Background subtraction

Acquired data contain some degree of background noise. The background subtraction process subtracts the background spectrum (normally, the spectrum at the chromatogram baseline) from the specified measured spectrum (normally, the spectrum in the vicinity of the peak apex), resulting in a single spectrum. This is performed prior to qualitative analysis.

Band identification method

See Time Band method.

Base peak

In a mass spectrum, the relative intensity of each ion is normally found using the peak with the highest intensity as the "standard" or "base" peak.

Batch processing

(1) Each line in the batch table contains injection and analytical parameters for that sample. After performing sequential analysis of the samples, data processing and report printing are automatically performed.

(2) Re-processing of previously acquired data can also be performed with the instrument off-line.

Blank nut

This is a GC accessory used to seal a flow line, such as the carrier gas or split/purge lines. It is also referred to as a blind nut.

C

Calibration curve

This is a curve showing the relationship between the compound concentration and the actual area or height obtained for that compound during analysis. In the GCMSsolution software, the horizontal axis represents the concentration of the compound and the vertical axis represents the area or height obtained by analyzing the sample. The resulting curve is used to quantitate unknown samples.

Capillary column

A capillary column is made of quartz glass capillary tubing; the inner surface is coated with a chemically bonded stationary liquid phase, and the outer surface is coated with a polyamide resin to prevent breakage. The column separates the components of a sample injected into the GC injection port. Except for samples injected with a DI (optional), the column separates all samples.

In its standard configuration, the GCMS-QP2010 accepts columns with an inner diameter of less than 0.32 mm. The maximum carrier gas flow rate in the GCMS-QP2010 is 15 mL/min, enabling direct connection of a 0.53 mm column to the MS.

CI (Chemical Ionization)

This is an ionization method where reagent gas reacts with the sample molecule to produce ions. Fewer peaks are produced compared to EI, but since it is a "soft" ionization method (with a complex ionization mechanism), it has the advantage of being able to obtain molecular weight information for compounds that cannot be analyzed by EI.

When detecting negative ions, this method is called negative ion chemical ionization (NCI).

Channel

See group.

Chromatogram

This is the graphic output (plot) obtained by detecting compounds as they elute from a chromatograph.

Condensation

Water droplets can form and adhere to the surface and interior of the instrument. Since condensation is damaging to the instrument, exercise caution during installation.

Compound Finder

Checks to see if the target compound is in the sample matrix. Because it uses spectrum similarity, it is necessary that the target compound standard spectrum be recorded in the compound table.

Consumable parts

These are parts that wear out during use of the GCMS-QP2010, e.g., the GC injection port septum or ion source filament. Maintain adequate supplies of these parts.

Conversion Dynode

The conversion dynode is located at the entrance to the detector. A high voltage electrode, it efficiently converts positive ions into electrons or negative ions into positive ions, according to polarity. It increases sensitivity, particularly in the high mass range, and facilitates the detection of negative ions. It is used as the detector in the GCMS-QP2010.

Corrected area normalization method

This method quantitates each peak according to an external standard calibration curve, then adds the quantitation values and determines the percentage of the quantitation values for each component with respect to this total. See Appendix A "Peak Processing and Mass Spectrum Operations" on page 273 for more information.

Corrected area normalization with scale factor method

This method calculates peak concentrations using the total area (height) as a scale factor (dilution factor), rather than letting that total be 100 (concentrations expressed as a percent of total.) See Appendix A "Peak Processing and Mass Spectrum Operations" on page 273 for more information.

D

Database

See library.

Deflector

See off-axis deflector.

Detector

This component converts measured sample characteristics into an electrical signal. The sample is ionized and passes through the ion source box. The ions are mass-separated by the quadrupole rods and then converted into an electrical signal by the detector. The GCMS-QP2010 uses an electron multiplier equipped with a conversion dynode.

DI (Direct Sample Introduction)

The sample is inserted directly into the MS with a DI probe. The probe is heated to vaporize the sample, which is not separated by the GC column.

E

EI (Electron Impact Ionization)

This common method obtains ions from sample molecules. Electrons from a filament are accelerated to 70eV and directed into an ionization chamber, where they impact with the sample molecules present. Because this is a "hard" ionization method, the molecules are cleaved and fragments are produced. Structural information can be obtained from the fragments, and a database of spectra makes library searches possible. This is a highly reproducible and reliable method for qualitative and quantitative analysis.

Electron Volts (eV)

This is the energy applied to the electrons for ionizing a molecule or atom.

Emission current

The filament is heated by passing a relatively large current through it. Current in excess of the heating current causes the release of electrons by thermal emission. This emission current is very small in comparison to the heating current (approx. 75 mA), and is controlled electronically so that its amperage is constant. The majority of the electrons move towards a trap or collector electrode from the ion source box. The collector current is always less than the emission current.

Evacuation rate

This expresses the evacuation capability of a vacuum pump.

Event

The acquisition mode is set for the Event.
Up to two events can be set for each group. Set the Acquisition mode for Event 1 to Scan, and SIM for Event 2.
If two events have been set, the start and end time in successive two rows for the SIM table (MS parameter) should be set to the same values respectively.

Event Time

This is the acquisition interval for each event. If only one event is set for a group, the Event Time is the same as the acquisition interval. If two events have been set for a group, the sum of the Event Time for Scan mode and SIM mode becomes the acquisition interval for the group.

External standard method (absolute calibration curve method)

This quantitative method calculates the concentration of a target compound by creating a calibration curve showing the relationship between the absolute mass or concentration of a compound in a standard and the area or height of its peak. An unknown sample is analyzed under identical conditions, and the calibration curve is applied to the area or height of the peak in the unknown sample. For this method, the analytical conditions of the unknown sample must be exactly the same as those of the standard. Injection volumes must be constant, since the method accuracy is dependent on the volume of the sample injected. See Appendix A "Peak Processing and Mass Spectrum Operations" on page 273 for more information.

F

Feed-through

Terminal where electrical signals enter without compromising the vacuum in the MS.

Filament

The filament is made of rhenium. Connected to the ion source box, it generates the electron beam necessary for ionizing the samples. Two filaments are present so that if there is a problem with one of the filaments, the operator can switch to the other one.

File

In computer terminology, a compilation of data into a single unit is referred to as a file. In the GCMSsolution software, the data, tuning conditions, methods, batch schedule, report format, and libraries are each compiled into files, and are saved with the extensions shown below.
Data file (*.QGD)

Tuning file (*.QGT)

Method file (*.QGM)

Batch schedule file (*.QGB)

Report format file (*.QGR)

Library file (*.LIB)

Format (report format)

In the GCMSsolution software, various types of reports can be printed, including the method, data processing results or library search results. Format refers to the customizable layout of these items.

G

GC pressure parameters

The pressure parameters precisely control the pressure of carrier gas flowing through the GC column at each stage of an analysis.

GC temperature parameters

The temperature parameters precisely control the temperature of the column oven at each stage of an analysis. Injection port and interface temperatures can also be specified.

Ground

For safety, ensure that the GCMS-QP2010 is properly grounded to an earth ground during operation.

Group (channel)

This consists of the mass of ions to be measured during a time interval of a SIM analysis. A different group is normally used for each compound present.

A single analysis can be grouped into periods of time. The time bands are referred to as groups or channels. There can be up to 128 groups; up to 64 masses (ions) can be set for each group. Therefore, up to 128 × 64 or 8,192 masses (ions) can be monitored.

I

Index search

This search method extracts information from a specified library file on the basis of a specified search index, or list of criteria for filtering searched information.

Interface

See Transfer line interface.

Internal standard method

An internal standard compound is added to each standard and sample. First, a calibration curve is created, expressing the relative sensitivity and mass ratio of a standard target peak relative to the internal standard peak. The concentration of the target compounds is then calculated by applying this calibration curve to the area ratio or height ratio of the peak of the unknown sample. This method compensates for variations in analytical conditions.

Ionization

This is the process of removing one or more electrons from a molecule or atom and converting it to a positive ion. In the GCMS-QP2010, the column separates the injected sample, after which it is ionized by the ion source. Although EI is used as the standard ionization method, CI and NCI ionization are other ionization options.

Ion source

This component ionizes the eluted compound from the column. It consists of the ion source and Lens 1.

Ion source box

The Ion source box consists of the ion source and lens stack. It is used to ionize the sample molecules and direct those ions to the quadrupoles.

L

Lens stack

Lens 2, Lens 3 and Lens 4 make up the lens stack, which efficiently extracts and accelerates the ions produced by the ion source, directing them to the quadrupole rods.

Level

Up to 64 calibration points can be specified for a calibration curve in the GCMSsolution software. Level 1 through 64 is assigned to each of these calibration points. Each level can be determined by calculating the average of up to 10 repeat injections.

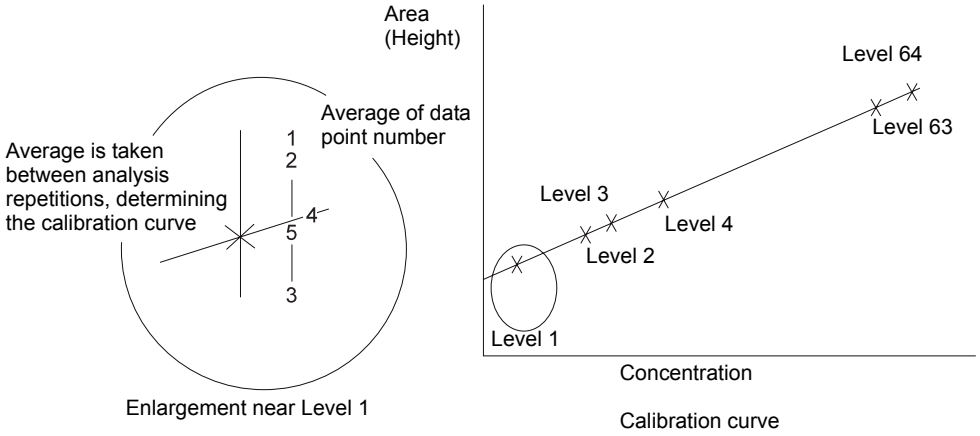


Figure F.1 Calibration Levels

Library (library file)

This is a compilation of mass spectral data used for similarity searches. Library files include public library files, such as the NIST database, and private library files that can be created by individual users. When a similarity search is executed, similar mass spectra are sought from the specified library files.

Library search (similarity search)

The mass spectrum of an unknown compound is compared to the spectra in a library file (database); the most similar spectra from the library file are listed. In addition, mass spectra can be selected from library files using specific search indexes (index search).

Linear

This is a calibration curve fit type for quantitation. Up to 64 different standard concentrations are analyzed, and a linear calibration curve is calculated by the least squares method. If there is only a single calibration level, a simple straight line passes through the single point and the origin. If there are two or more calibration levels, and the curve does not pass through the origin, a linear calibration curve is drawn through those two points. In other cases, a linear curve is drawn using the least squares method. Each point can represent the average of up to 10 individual analyses.

M

Main rod

See quadrupole rods.

Maintenance parts

These are parts that are necessary for the safe and optimal operation of the GCMS-QP2010. These parts are not changed frequently, but should be changed as they deteriorate.

Mass number

This refers to the integer value in atomic mass units of an ion within a mass spectrum. The mass analyzer measures the exact mass of an ion, but the mass number represents the closest integer to that the exact mass.

Mass calibration

Using a calibration standard, such as PFTBA, a spectrum is acquired. During the scan of the PFTBA spectrum, the exact time of detection of a known mass peak is matched with the mass peak's exact mass. Approximately 20 exact mass values of PFTBA are matched with the exact time of detection during a single spectrum scan. Since the mass analyzer can only measure the time during a spectrum scan, it uses mass calibration to identify the mass using the times at which mass peaks are detected.

Mass pattern adjustment

In the GCMS-QP2010, the mass pattern can be adjusted to any pattern (intensity ratio) using the Tuning application.

The mass pattern adjustment refers to the process of adjusting a mass spectrum (raw data) acquired by a quadrupole mass spectrometer using the mass patterns from a magnetic field mass spectrometer as a standard. Otherwise, the patterns acquired with a quadrupole mass spectrometer would differ from those acquired with a magnetic field mass spectrometer, even though the mass spectra (raw data) are identical. The mass pattern from a magnetic field mass spectrometer is used as the standard because of the vast amount of data accumulated and used over its long history.

Mass spectrum

This refers to the bar graph display of the intensities of ions in their m/z order.

Megabore column

Name used for a capillary column of diameter 0.5 - 0.53 mm.

Method

This refers to a compilation of the parameters controlling the MS and GC units for data acquisition and data processing. Methods are saved as method files. The advantages of using methods are listed below.

(1) When analyzing samples similar to those analyzed in the past, analyses can be performed with a previously developed method, without having to reset the various parameters.

(2) By using a Batch Table, automated analyses can be performed using a different method for each sample in the autosampler.

(3) Since analysis parameters and data processing parameters can be saved as a single file, data management is simplified.

Method file

This is a file used to save a method that has been developed in the "Analysis" window or in Method Development mode. It is saved with the extension .qgm.

Molecular formula

This is a chemical formula that shows the actual numbers and types of atoms in a molecule, but not the chemical structure.

Molecular ion

A molecular ion is formed from the loss of one electron from a molecule, without breaking any intramolecular bonds. Since many compounds have isotopic atoms, the molecular weight of a pure compound and the m/z value of the molecular ion peak are defined as the most abundant isotope peak found at the highest value of m/z in the spectrum. For benzene, which has substantial m/z 79 and m/z 80 peaks, the molecular ion is considered to be at mass 78.

Multiple reference relative retention time method

When large retention time shifts occur, retention time correction can be improved by selecting multiple reference peaks. In this method, the peaks are classified into multiple zones. A reference peak is specified for each zone, and the peaks within the zone are identified based on each zone's reference peak.

See also Relative retention time method.

m/z (mass-to-charge ratio)

This is a value that divides the mass of an ion by its electrical charge number.

N

Narrow bore capillary column

This is the general term for capillary columns with an inside diameter of 0.2 - 0.25 mm. This is the standard column used in the GCMS-QP2010.

NCI (Negative ion Chemical Ionization)

See CI (Chemical Ionization)

Needle valve

This is a valve that makes minute flow regulation possible.

NIST

This is the acronym for the National Institute of Standards and Technology (US).

Normalization method (Area normalization)

The detected peak areas or heights are added, and the percentage of each peak area or height with respect to the total value is determined. See also Corrected Area Normalization method.

O

Off-axis deflector

The deflector electrode produces a negative charge, which helps to direct ions to the detector. It is off-center with respect to the central axis of the quadrupoles.

O-ring

This is a rubber ring that is installed at the interface between certain types of connections, for example, at the contact surfaces between the door and the analyzer housing. The O-ring seals the two surfaces and prevents vacuum leaks.

OS (Operating System)

This underlying software program efficiently manages the overall computer operation and resources. All of the software for the GCMS-QP2010 operates under MS-Windows 2000, which is one type of OS.

P

Peak profile

This refers to the raw peak of a mass spectrum, displayed in the "Peak Monitor" window. In scan analysis, a mass spectrum is obtained by processing this raw peak.

Peak report

In the GCMSsolution software, the peak processing results and quantitation results for analyzed data can be compiled and displayed and/or printed out. This compiled table is called the peak report.

PFTBA

This is the acronym for perfluorotributylamine. PFTBA is one of the tuning standards normally used for mass analysis when ions with a mass of up to 700 are analyzed. It is used when performing resolution adjustment, sensitivity adjustment (mass pattern or target mass), and mass calibration.

Molecular formula: $(C_4F_9)_3N$

Point-to-point calibration curve

This is a calibration curve fit type for quantitation. Up to 64 different standard concentrations are analyzed, and a point-to-point calibration curve is created. No curve fit constant is displayed in the quantitation table when a point-to-point curve fit is specified, and none can be entered. If there is only a single calibration level, a simple line that passes through that point and the origin is drawn. If there are two or more calibration levels, a point-to-point curve is drawn; the initial line must pass through the origin. Each point can represent the average of up to 10 individual analyses.

Pre-amp

The pre-amp amplifies the output current from the secondary electron multiplier.

Pre-rod

See quadrupole rods.

Private library

See library.

Pt sensor

Platinum is used as a temperature sensor because its resistance is dependent on the temperature. For example, a Pt sensor is used to control the transfer line interface temperature.

Purge vent

This is the carrier gas outlet of the GC-2010 purge line. Connect a bubble flow meter to this outlet to check the purge flow rate.

Q

Quadratic/cubic

This is a calibration curve fit type for quantitation. Up to 64 different standard concentrations are analyzed, and a second order (quadratic) or third order (cubic) curve is drawn by the least squares method. For quadratic curves, three or more calibration curves are required. For cubic curve fits, four or more calibration points are required. The curve will be calculated as a quadratic equation if there are three points and as linear if there are two or fewer points. Each point can represent the average of up to 10 individual analyses.

Quadrupole mass filter

An electromagnetic field is obtained by applying RF amplitude and DC voltage to the quadrupole rods, creating a mass filter that allows passage of only the ions with a specified mass.

Quadrupole mass spectrometer

This consists of an ion source, four quadrupole rods, and a detector, as shown in the figure below. As the ions pass through the quadrupoles, they are separated by mass.

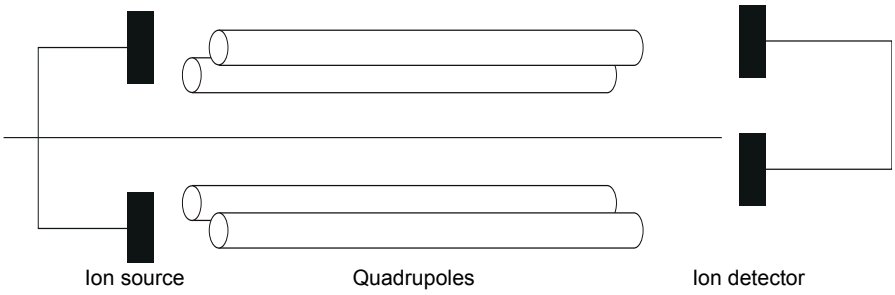


Figure F.2 Quadrupole Mass Spectrometer Diagram

Quadrupole rods

The four quadrupole rods operate in rod pairs to form a hyperbolic electrical field. The main rods have both RF and DC voltage applied.

The GCMS-QP2010 also uses pre-rods, where only RF voltage is applied. This keeps the main quadrupoles clean and improves ion transmission.

Quantitation

This refers to the process of determining the concentration of a compound or the concentration ratio of the compound in a sample. Quantitation in the GCMSsolution software can be performed by several calculation methods.

R

Reference

See Type (Type selected in Method).

Reference ion

Reference ion intensity ratios can be used along with retention time for compound identification. Up to 5 reference ions can be specified for comparison to the base peak in the compound spectra. For a compound to be identified, each reference ion must be within an specified range of relative intensity compared to the target ion.

Relative retention time method

In this method, identification is performed after correcting for the retention time drift for each peak. First, a specified reference peak is identified by the absolute retention time method; then the target peaks are identified by the relative retention time. Specifically, the actual retention time of the target peak is corrected by calculating a ratio based on the shift in retention time of the reference peak.

See also Multiple reference relative retention time method.

Repeller Electrode

Electrode that forces ions out of the ion source box.

Resolution

Resolution expresses the degree to which two adjacent peaks are separated. Resolution adjustment refers to adjusting the peak width of mass peaks. The resolution when analysis is performed by a quadrupole MS is shown by the following formula.

$$R = \frac{W}{\Delta W} \times \frac{M}{\Delta M}$$

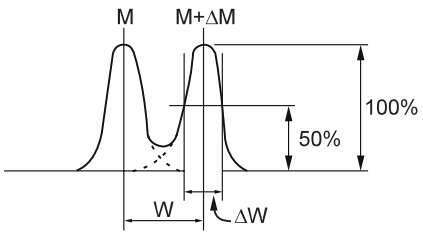
R: Resolution

M: Mass of measured peak

ΔM: Mass difference between measured peak and adjacent peak

W: Interval between measured peak and adjacent peak

ΔW: Measured peak width



For a quadrupole MS, the following expression is used.

$$\frac{W}{\Delta W} \times \frac{1}{\Delta M} \times M$$

In cases where the above expression is equal to 2, subsequent multiples of the peak height can be expressed as:

$$\frac{W}{\Delta W} \times \frac{1}{\Delta M} = 2M$$

RF power supply

This is the power supply that generates the high frequency voltage and direct current voltage applied to the quadrupoles.

Rotary pump

Also referred to as a rotary vane vacuum pump. It produces a vacuum of 10^{-3} Torr and is used as the backing pump to the turbomolecular pump. In the GCMS-QP2010, the standard configuration uses one rotary pump, but if a DI or CI unit is added as an option, an additional rotary pump is required.

See also Evacuation rate.

S

Sample type

(Set in Batch Table and Sample Log-in)

The samples are categorized by type according to the analysis objectives, as follows:

Unknown: Sample of unknown content and concentration. Concentration calculations are performed using a previously acquired calibration curve.

Standard: An injection of a standard solution of known concentration; used to create a calibration curve. The manner in which points are added to the calibration curve is also selected.

Initialize calibration curve:
All of the previous calibration curve information in a method is cleared. Ordinarily, this is selected for the first standard in a batch table.

Add calibration level:
A calibration point is generated at the level indicated by the level number column. If there is already a point at this level, then the value is averaged with the previous results.

Replace calibration level:
A calibration point is generated at the level indicated by the level number column. If there is already a point at this level, then the previous results are deleted and a new point is generated.

Control: Standard solution or QA/QC sample designated as control in the properties section of the data file. Processing is the same as with an unknown sample.

Unspiked: Unspiked unknown sample in an unspiked/spiked pair.

Spiked: An unknown sample is spiked with a known amount of standard. The percent recovery of the spike is calculated using the unspiked sample, which is analyzed immediately prior to the spiked sample.

Standard (ISTD Recovery): Used when the QA/QC function requires the ISTD % recovery to be calculated.

Savitzky-Golay method

Digital filter for smoothing that uses the least squares method. In comparison to the moving average method, it allows high-frequency signal components to be more easily transmitted, but has inferior noise performance.

Scan

This is the process of analyzing mass spectra by continuously changing the RF voltage or DC voltage in one direction as applied to the quadrupoles.

The interval of the change is called the Event Time and the rate of the change is called the scan speed. If the range of masses 10 to 610 were scanned at an Event Time of 0.1 second, the scan speed would be 6,000 AMU/second.

Scan speed

See scan.

Search index

This refers to one more criteria used for performing an index search. In the GCMSsolution software, the molecular weight, molecular formula, class, retention index, base peak and compound name can be used as a search index.

Sensitivity adjustment

This consists of adjusting the ion source lens voltages, RF parameters and detector voltage to obtain the best intensity and peak shape for a specific PFTBA mass peak chosen in "Edit Tuning Condition" dialog box in Auto Tuning.

Septum purge

This is a flow path, located just under the septum in the injection port, that uses carrier gas to sweep any contaminants from the injection port and reduces or eliminates large tailing solvent peaks.

Silica capillary

This is a capillary column in which the fused silica base is coated with polyamide resin as the stationary phase. See also Capillary Column. It is used as resistance tubing to control the amount of PFTBA tuning standard introduced.

SIM (Selected Ion Monitoring)

This method detects only specific masses. As with MC, chromatograms are obtained for each mass. The ions to be detected are specified prior to analysis. Since the number of ions detected is limited, detection sensitivity is extremely high, making it possible to measure picogram quantities of compounds.

Similarity search

See Library Search.

Solvent flush method

In this injection method, sample, air and then solvent are injected in that order, by drawing them into the syringe in the opposite order, as shown in the figure below. Solvent flush injection of samples can be performed reliably by a variety of methods with the AOC autosampler.

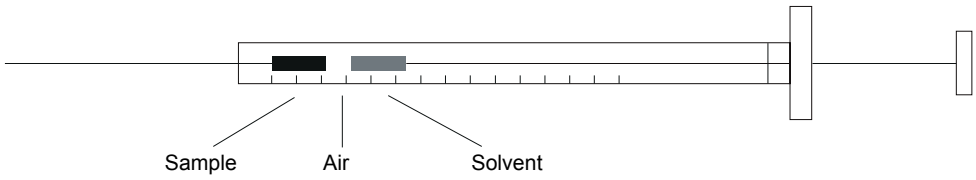


Figure F.3 Solvent Flush Method

Split injection

This is a sample introduction technique used primarily in capillary chromatography when sample concentrations are high. The sample is introduced into the injection port, where it is divided into two flow paths. One flow path enters the analytical column, while the other is vented.

Splitless injection

This is a sample introduction technique used primarily in capillary chromatography when sample concentrations are low. Sample is introduced into a hot split/splitless injection port, where the solvent is vaporized, but not the analytes. A small amount of solvent flows into the relatively cool column and recondenses. The analyte compounds collect in the recondensed solvent located at the head of the column. After a period of time has elapsed (the sampling time), the split vent opens and sweeps the excess solvent vapor out of the injection port. A temperature program then begins in the column oven.

Split vent

This is the carrier gas outlet of the split line flow path. Connect a bubble flow meter to this outlet to check the split flow rate.

Standard Addition

Coexisting compounds in the sample sometimes can skew the concentration results of a target compound. This is referred to as the matrix effect. This calibration curve method compensates for this adding various levels of a known standard to identical quantities of sample and calculating the concentrations of the samples. In other words, a sample with no spiked standard, and the same sample with various levels of standard added are analyzed. A calibration curve is created with the quantity of standard added on the horizontal axis, and the peak area or height on the vertical axis, and quantitation is performed.

T

Target

See Type (Type selected for Method).

Temperature control zones (heated zones)

These are components of the system with temperature control.

TIC (Total Ion Chromatogram)

The TIC represents the sum of the ion intensities at a particular time. The TIC is similar to chromatograms detected by gas chromatography using a FID (Flame Ionization Detector) detector.

TIME BAND method

This method uses a specific retention time span to identify each compound.

Allowable retention time band = +/-TIME BAND (minutes)

If the expected retention time is 5.0 minutes and the retention time band is 0.5 minutes, the allowable retention time band is 5.0 minutes +/-0.5 minutes, or from 4.5 minutes to 5.5 minutes. The advantage of this method is that an optimal retention time band can be specified for each peak. This method can be time-consuming to set up, as it is necessary to make the setting for each peak.

The Band method is useful when peak widths do not change over time, as in a GC temperature-programmed analysis.

TIME WINDOW method

This is a method specifies a certain retention time span for all peaks that is a percentage of the retention time. This window increases with retention time.

Allowable time window = +/-(Expected retention time (min) x TIME WINDOW (%))

If the expected retention time is 5.0 minutes and the time window is 10 %, the allowable time window is 5.0 minutes +/-10 % of 5.0 minutes, or from 4.5 minutes to 5.5 minutes. This method is simple to set up, but has the disadvantage that the window allowed for each component can overlap with the windows of other components.

This method is used for analyses where the peak widths increase with time, as in isothermal analysis.

Transfer line interface

This component connects the capillary column to the MS ion source.

Triazine

Abbreviation for tris(perfluoroheptyl)-S-triazine. Used when mass calibration or sensitivity adjustment is to be performed for ions of mass 1000 or less. Triazine is commercially available.

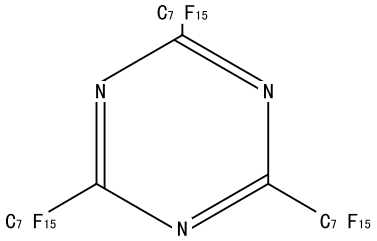


Figure F.4 Triazine Structural Formula

Type (Type selected in Method)

When performing identification and quantitation, the standard compounds are categorized into the following types according to the way they are used.

Target: This is a compound that is the object of quantitation.

Internal Standard: This is a compound of known concentration added to all standards and samples for the purpose of quantitating other components in the sample. Also referred to as IS.

Reference: This is a known compound added to all standards and samples for the purpose of identifying other components in the sample. The expected retention time and actual retention time of the reference peak is used to relatively correct for any retention time shifts. Up to 8 reference compounds can be designated.

Internal Standard & Reference: An internal standard compound can also be used as a reference for retention time correction.

Discovery: Select this when using the Compound Finder.

Tuning

Tuning is the process of optimizing the MS. Tuning of the MS is performed in order to:

(1) Verify that there are no abnormalities in the status of the instrument, and trace the cause of any problems.

(2) Determine the optimum operation parameters.

The types of adjustments include mass calibration, resolution (peak width) adjustment and intensity adjustment. Intensity adjustment can be performed by sensitivity adjustment, either by specifying a target mass, or by relative intensity adjustment (mass pattern adjustment).

Turbomolecular pump

This vacuum pump maintains a high vacuum in the ion source and analyzer within the vacuum housing. Gas molecules collide with the moving rotor blades, directing the gas molecules in a certain direction. If the gas molecules collide with other gas molecules more often than the rotor, then the pump stops. Therefore, the turbo pump must be backed up by another type of pump, usually a rotary pump.

See also Evacuation rate.

V

Version upgrade

This refers to a software improvement. Normally, when a version is upgraded, a new version number is assigned.

The current version number of the software can be checked by selecting the About GCMSsolution command in the Help menu of the GCMSsolution software.

Vespel ferrule

This is a ferrule made from polyamide resin that seals the capillary column interface.

W

Wide bore capillary column

Designation for capillary columns of inner diameter 0.32 mm. Can be used in the standard GCMS-QP2010 configuration.

Window identification method

See Time Window method.

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