638-94247A
(M393-E200)

Total Organic Carbon Analyzer

TOC-VWP &
TOC-Control V Software

User Manual

Read this manual carefully and keep it with the instrument for future reference.

SHIMADZU CORPORATION

ANALYTICAL & MEASURING INSTRUMENTS DIVISION
KYOTO, JAPAN

Read and understand this manual before operation. Save this manual.

Information in this publication is subject to change without notice and does not represent a guarantee on the part of the vendor.

Any errors or omissions which may have occurred in this publication despite the utmost care taken in its production will be corrected as soon as possible, but not necessarily immediately upon detection. We appreciate notification of any errors or omissions.

Shimadzu does not have any responsibility for errors or injuries resulting from following the instructions in this manual.

Introduction

Thank you for purchasing the TOC-VWP Total Organic Carbon Analyzer.

This instruction manual is a TOC-V user manual. It provides operation and maintenance procedures for the TOC-V. The procedures related to TOC-Control V software system administration are described in detail in the TOC-V Administrator's Manual.

Be sure to carefully read this documentation before using the instrument for the first time. Read the Operational Precautions before operating the instrument. Follow the procedures in the manual to avoid compromising user or instrument safety.

CAUTION

- Do not operate this instrument before understanding the contents of this manual.
- If this manual or the warning labels on the instrument become lost or damaged, promptly obtain replacements from your Shimadzu representative.
- To ensure safe operation, read and follow the procedures in Section 1.2 "Operational Precautions" before operating the instrument.

Product Warranty and Post Sale Service

Product Warranty

The warranty does not cover malfunctions that result from:

- misuse;
- repairs or modifications made by any company other than the manufacturer or an approved company;
- external factors;
- operation under severe conditions, such as environments with high temperature, high humidity, corrosive gas, vibration, etc.;
- fire, earthquake or other forces of nature;
- moving or transporting the unit after its initial installation;
- the consumption of items or parts that can be regarded as consumable.

Post Sale Service

If any problems occur with this instrument, inspect it and take appropriate corrective action as described in Section 5.6 “Troubleshooting”. If the problem persists, or symptoms are not covered in the Troubleshooting section, contact your Shimadzu representative.

Contents of This Manual

Chapter 1 "Overview"

This chapter provides an overview of the instrument and operational precautions for its use.

Chapter 2 "System Description"

This chapter describes the TOC-V system components and optional accessories.

Chapter 3 "Software Overview, Administration, and Setup"

This chapter provides an overview of the TOC-Control V software and describes the software's Administration and Instrument Setup Wizard tools, which are used to manage user accounts and configure new systems.

Chapter 4 "Operation"

This chapter describes pre-analysis preparations and instrument and software functions, such as setting measurement parameters, starting up the instrument, generating calibration curves, and conducting sample analysis. This chapter also includes a tutorial that covers setup and measurement procedures.

Chapter 5 "Maintenance"

This chapter describes maintenance procedures and periodic checks required to ensure that the instrument provides consistently high accuracy analyses. Troubleshooting procedures and error messages are also included in this chapter.

Chapter 6 "Reference Materials"

This chapter describes principles of analysis and lists specifications, standard and optional accessories, and specialized terms associated with this instrument. This chapter also includes the installation procedure, for use in the event that instrument is moved to another site.

Appendix A "Method Validation"

This appendix describes the software's Method Validation tool, which is used to test the variance and linearity of a data set.

Appendix B "Control Charts"

This appendix describes the software's Control Charts tool, which is used to monitor the precision and accuracy of a data set.

Notation Conventions

Precautionary Conventions Used in this Manual

The following precautionary conventions are used in this manual.

Convention	Description
CAUTION	Indicates the possibility of slight to moderate injury or equipment damage.
Note:	Provides additional information to ensure the correct use of the instrument.

Other Conventions Used in this Manual

Supplementary Conventions

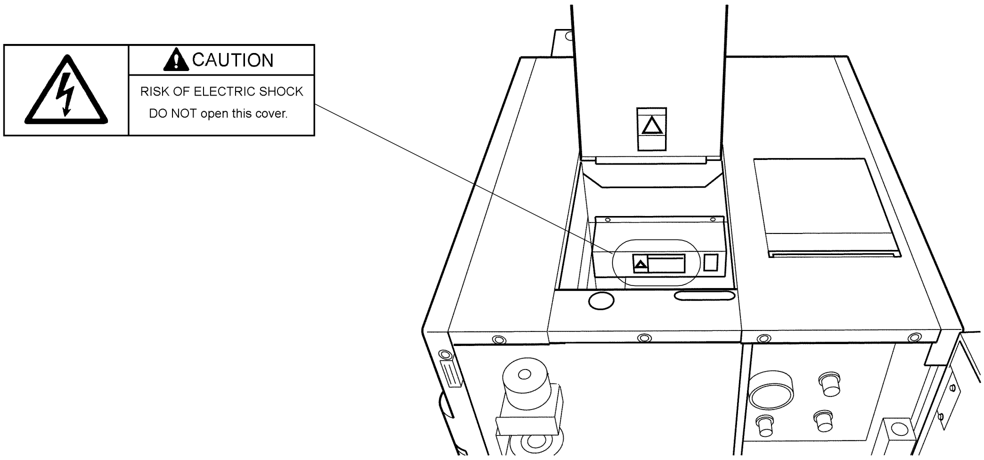
In addition to the precautionary conventions, Tip and Reference are used to supply additional information.

Convention	Description
TIP:	Indicates a particular technique, alternative means of operation, or useful advice.
Reference:	Indicates a reference location for more detailed information.

Warning Labels Used on this Instrument

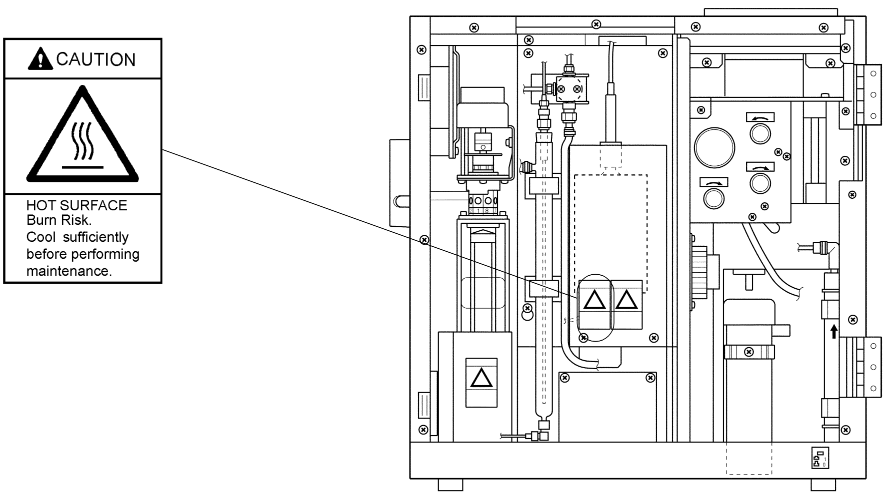
Electric Shock Warning

High voltage is applied to the UV lamp when it is illuminated. Never touch this component as there is a risk of electric shock. Always turn OFF the UV lamp before performing any UV lamp-related maintenance.



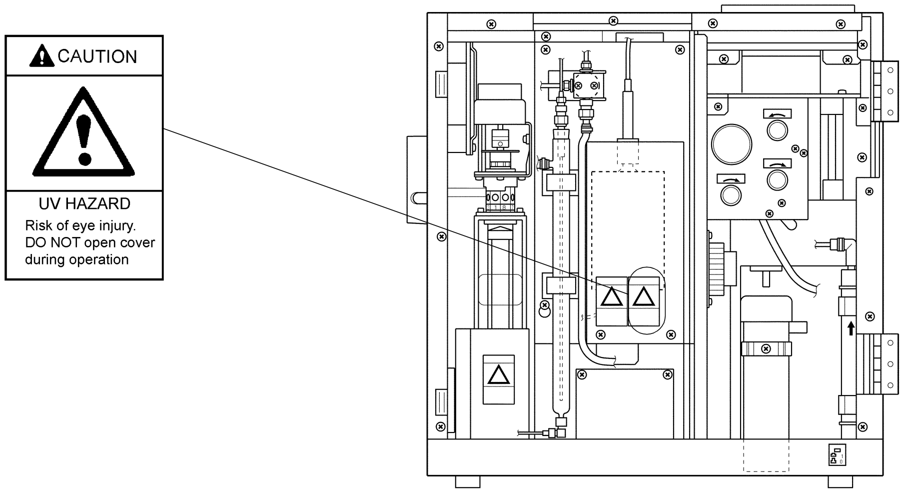
High Temperature Warning

The TC reactor and UV lamp are at high temperatures during operation (approximately 80°C). High voltage is applied to the heating element at the periphery of the aluminum block of the TC reactor heater. Never touch this area with bare hands as there is a risk of burn injury and electric shock. Turn OFF the TC reactor and UV lamp, and allow them to cool to room temperature before performing maintenance to the TC reactor or UV lamp.



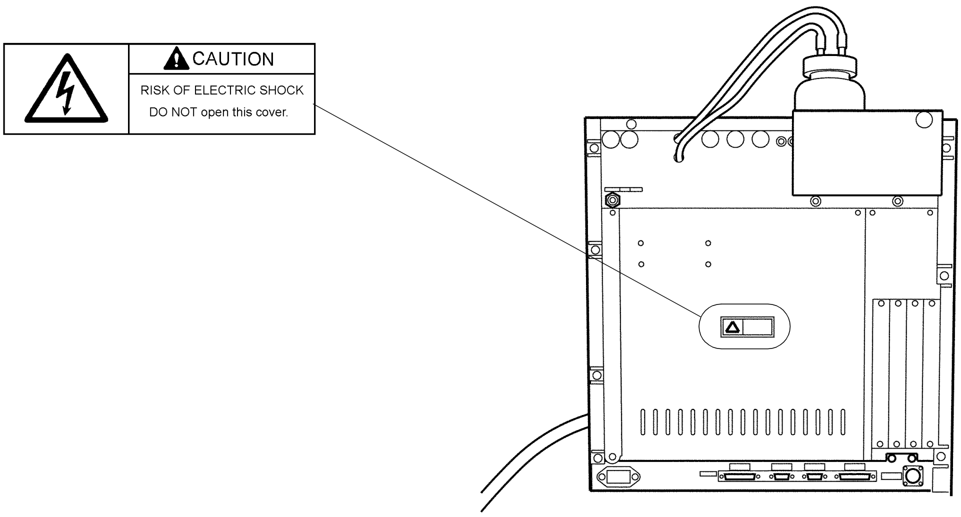
Ultraviolet Radiation Warning

Ultraviolet light is radiated from the UV lamp when it is illuminated. Due to the risk of eye injury, do not view this light directly for prolonged periods even with the supplied yellow cover in place. Before turning on the UV lamp, verify that it is correctly installed and that the supplied cover is attached.



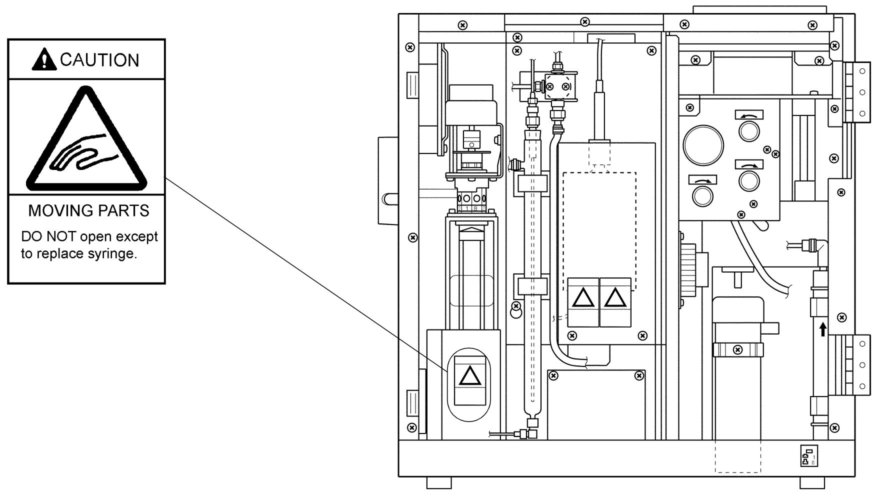
Electric Shock Warning

Do not remove this panel, as there is a danger of electric shock.



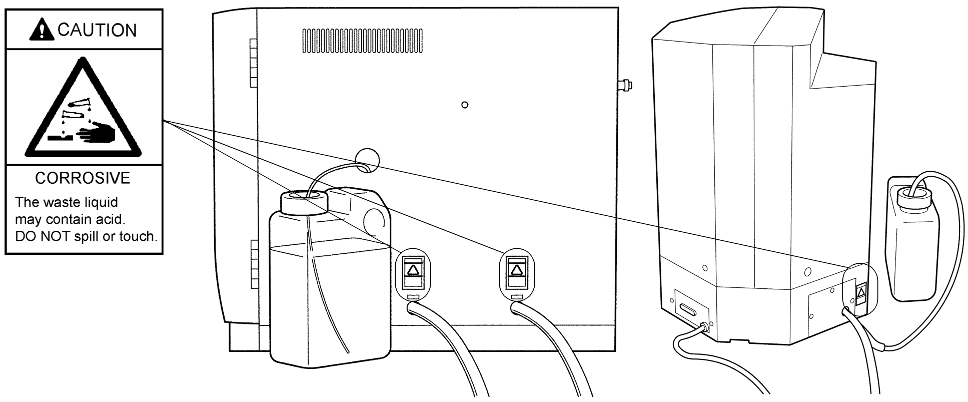
Injury Warning

The sample injection components of this instrument move during operation. To prevent injury, keep hands away from this mechanism.



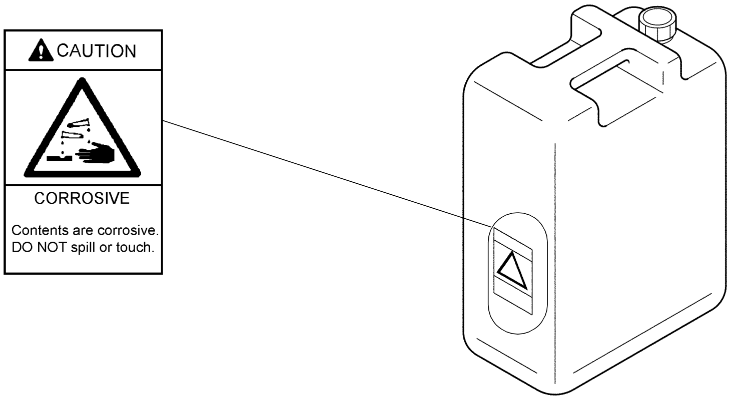
Corrosion Warning

Acid and other corrosive substances are present in the liquid waste drained from this instrument. Do not touch or spill this liquid waste.

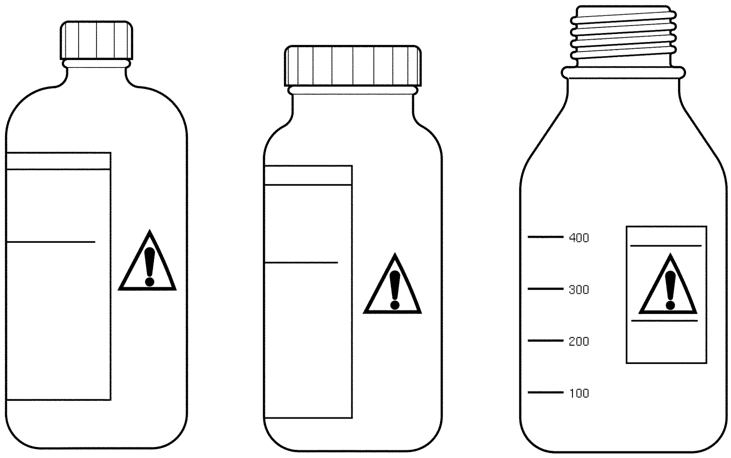


Corrosion Warning

The waste liquid contained in this receptacle is corrosive. DO NOT touch or spill this liquid waste.



Sodium persulfate (oxidizing reagent) and phosphoric acid are corrosive chemicals. DO NOT touch or spill these substances.



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1

Overview

This chapter provides an overview of the instrument and the operational precautions for instrument use.

1.1 Overview

The TOC-VWP is an instrument that measures the amount of total carbon (TC), inorganic carbon (IC) and total organic carbon (TOC) in water. “Wet chemical oxidation with non-dispersive infrared (NDIR) analysis” is a TOC measurement method that has been adopted by the JIS[1] and other international standards.

Moreover, when the ASI-V Autosampler or OCT-1 8-Port Sampler is used in combination with the main unit, a fully automatic system is created, allowing automatic analysis of multiple samples.

Read this user manual carefully before using TOC-VWP instrument to ensure its proper use. The manual should be stored in a convenient location for future reference.

⚠ CAUTION

Safety may be compromised if the instrument is used in a fashion other than that indicated in this user manual. Pay particular attention to the CAUTION sections to ensure safety when using the instrument.

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1. JIS K-0102 “Industrial Waste Water Testing”, JIS K-0551: “Total organic carbon (TOC) testing methods for ultra-pure water”, U.S.Pharmacopoeia, EPA 415.2, EPA 9060A, ASTM D2575, Standard Methods for Examination of Water and Waste Water 5310C

1.2 Operational Precautions

Take the following precautions when using this instrument.

CAUTION

- Ensure that the supplied cover for the UV lamp is installed. Do not view the UV lamp directly for prolonged periods even if the cover is installed. Ultraviolet radiation can cause eye injury. <Eye injury prevention>
- Ensure that the nitrogen carrier gas is flowing before turning on the UV lamp of the TC reactor. There is a danger of ozone gas generation, physical injury and corrosion of parts. <Physical injury prevention, part corrosion prevention>
- Ensure that the TC reactor cover is installed before turning on the reactor heater. There is a danger of burn injury due to the high temperatures at the TC reactor heater. High voltage is applied to the TC reactor heating element, creating a danger of electric shock. <Burn and electric shock prevention>
- The black rubber part at the top of the UV lamp reaches high temperatures when the UV lamp is illuminated. Do not touch this part of the UV lamp as there is a danger of burn injury. <Burn injury prevention>
- High voltage is supplied to the UV lamp when it is illuminated. Do not touch these parts. <Electric shock prevention>
- Use nitrogen gas as the carrier gas in this instrument. If air or oxygen is used, harmful ozone is generated inside the TC reactor, and could adversely affect health. <Prevention of harmful health effects>
- Turn off the UV lamp and allow the reactor to cool before performing maintenance to the TC reactor or UV lamp. Performing this maintenance while the UV lamp is on or the TC reactor is hot could result in burn injury or electric shock. <Burn injury and electric shock prevention>

- To reduce back pressure, verify that the external drain tubing connected to the waste port on the right side of the instrument does not touch the surface of the liquid in the waste container. The height of the external tube must always be lower than the height of the drain port. Excessive back pressure results in insufficient drainage and waste overflow inside instrument. <Part corrosion prevention>

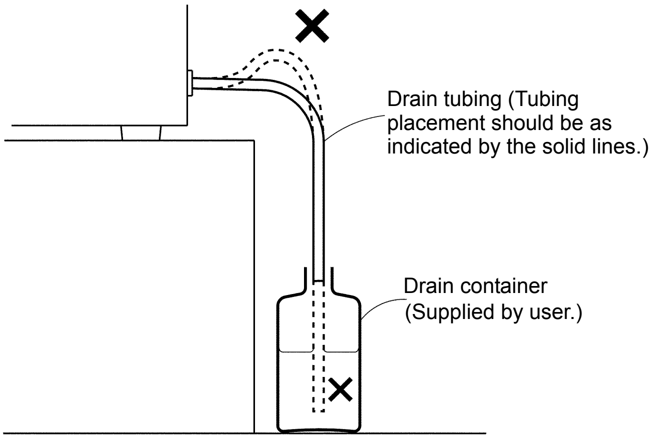


Figure 1.1 External Drain Tubing

- Hand tighten the connectors to the 8-port valve. Use of a tool may cause deformation and leakage in the valve. <Part damage prevention>
- Stop operation of the instrument when performing maintenance on any of the drive components, such as connecting lines to the 8-port valve or connecting/disconnecting lines on the sample injector. If these parts are manipulated or if the lines are disconnected during operation, the drive component could move, causing injury or discharge of liquid. <Injury prevention, corrosion prevention>
- This instrument is designed for measurement of relatively clean samples, containing almost no acid, alkali or salts. Depending on the concentration, the presence of these types of constituents could interfere with measurement accuracy.
- When measuring samples containing organic particulates (organic suspensions), insufficient oxidation could result in low values. In addition, slurries and other organic suspensions can accumulate in the instrument flow lines, damaging the Teflon parts inside the 8-port valve and the syringe adversely affecting instrument operation. It is recommended that such particles be removed from samples before measurement.
- The ASI-V autosampler can be equipped with an optional magnetic stirrer; however, this instrument is not designed for measurement of samples containing suspended particles. For this reason, references to the magnetic stirrer have been removed from this instruction manual.
- To clean the instrument, wipe the surface with a soft cloth moistened with water. Do not use chemicals.

- Repairs to the inside of this instrument can be dangerous. Contact your Shimadzu representative to have a trained service engineer perform any repairs.
- Do not disassemble or modify this instrument beyond the scope of the maintenance procedures in this manual as safety may be compromised.
- The construction of this instrument is not explosion-proof. It must not be used in dangerous areas.
- Use this instrument with a power supply of AC100-120V (for a 100V system) or AC220-240V (for a 200V system).
- Do not use the following characters when naming files or systems to be saved:

  / , . \ (back slash) : ; (these are all normal-width characters), space (normal width, em width)
- Turn OFF the Power Management function of the PC to prevent shutdown of the PC power while it is controlling the TOC analyzer. A shutdown of this nature would adversely affect performance.
- Hand-tighten the connectors to the 8-port valve of the 8-port sampler. Over-tightening with a tool may exert excessive force to the internal valve body, causing deformation and internal leakage. <Part damage prevention<
- Shut down the instrument before connecting or disconnecting tubing to the 8-port valve or performing maintenance to the drive parts. Liquid may be discharged from tubes during operation. <Part corrosion prevention>)
- When utilizing the software's printing function, set the paper size to letter or A4 and printer graphic resolution to 600 dpi.
- This instrument is equipped with a data backup battery with a life span of about 1-year. If the instrument is not turned ON at least once a year, it may not connect with the PC.

  If the battery is completely depleted, the instrument may not start up when it is first turned ON. If this occurs, leave the power ON for 30 seconds, then turn the power to the instrument off and back on again.

  If the instrument is to remain idle for a long period, turn the instrument ON and leave it running for 24 hours at least once a year.

1.2 Operational Precautions

2

System Description

This chapter identifies the components of the TOC-VWP, ASI-V autosampler and the OCT-1 8-port sampler.

2.1 TOC-VWP

2.1.1 Front View

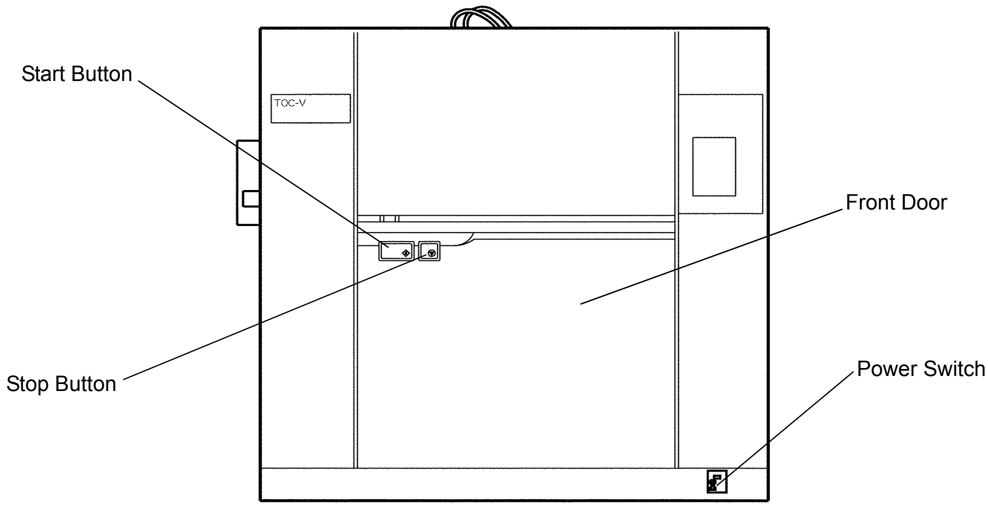


Figure 2.1 Front View

2.1.2 Right Side View

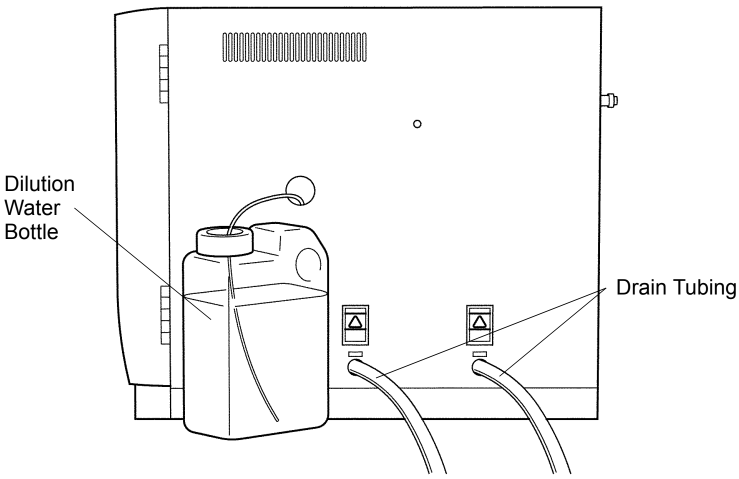


Figure 2.2 Right Side View

2.1.3 Left Side View

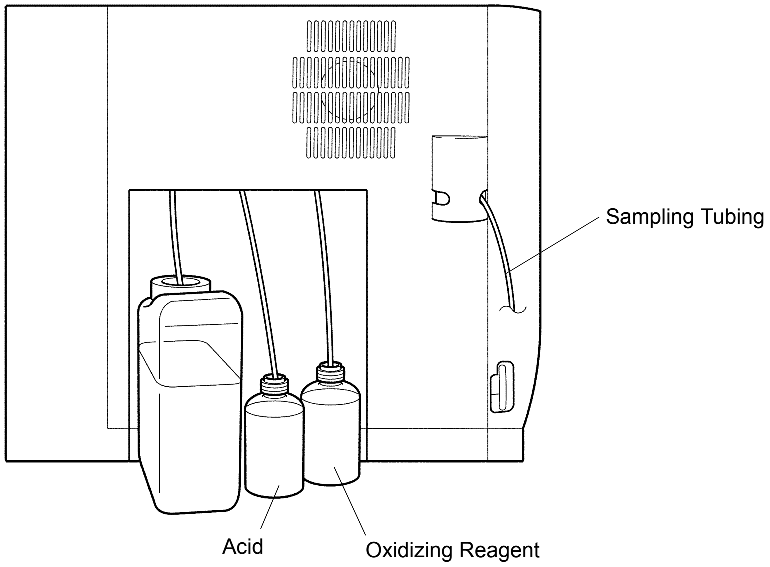


Figure 2.3 Left Side View

2.1.4 Rear View

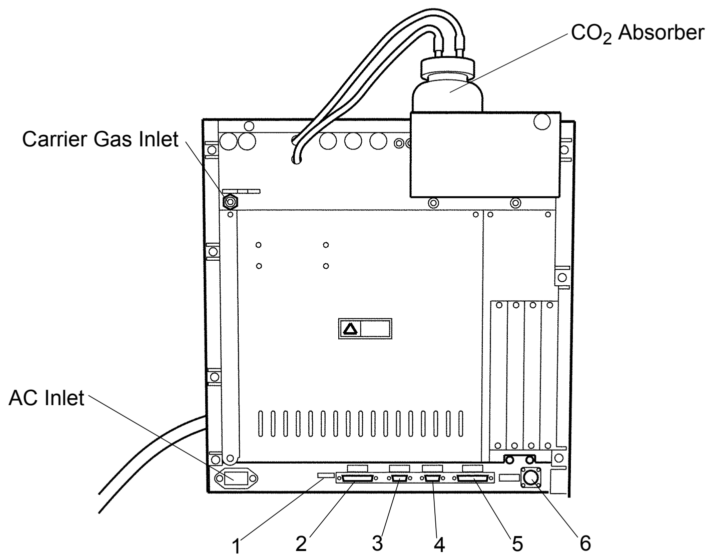


Figure 2.4 Rear View

1) Flash ROM rewrite switch
2) Printer cable terminal
3) RS-232C terminal (1)
4) RS-232C terminal (2)
5) ASI-V/OCT-1 signal cable terminal
6) ASI-V/OCT-1 power cable terminal

2.1.5 Inside Front View

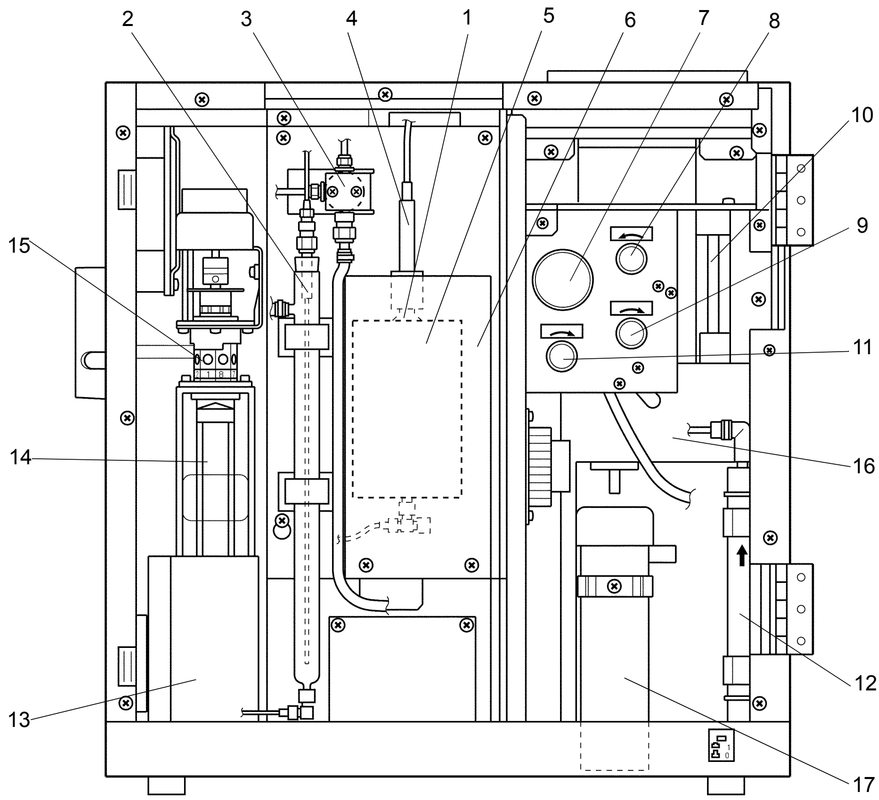


Figure 2.5 Inside Front View

1) TC reactor
2) IC reactor
3) Solenoid valve (for IC reactor gas switching)
4) UV lamp
5) TC reactor heater
6) TC reactor cover
7) Carrier gas/sparge gas pressure gauge
8) Carrier gas flow adjustment knob
9) Sparge gas flow adjustment knob
10) Carrier gas/sparge gas flow meter
11) Carrier gas/sparge gas pressure adjustment knob
12) Halogen scrubber
13) Syringe pump cover
14) 25mL syringe
15) 8-port valve
16) Dehumidifier
17) Dehumidifier drain pot

2.1.6 Top View

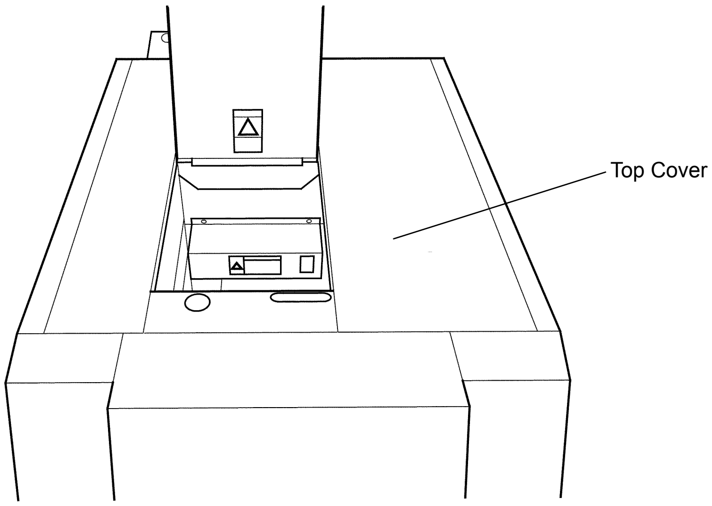


Figure 2.6 Top View

2.1.7 Flow Diagram (TOC-VWP)

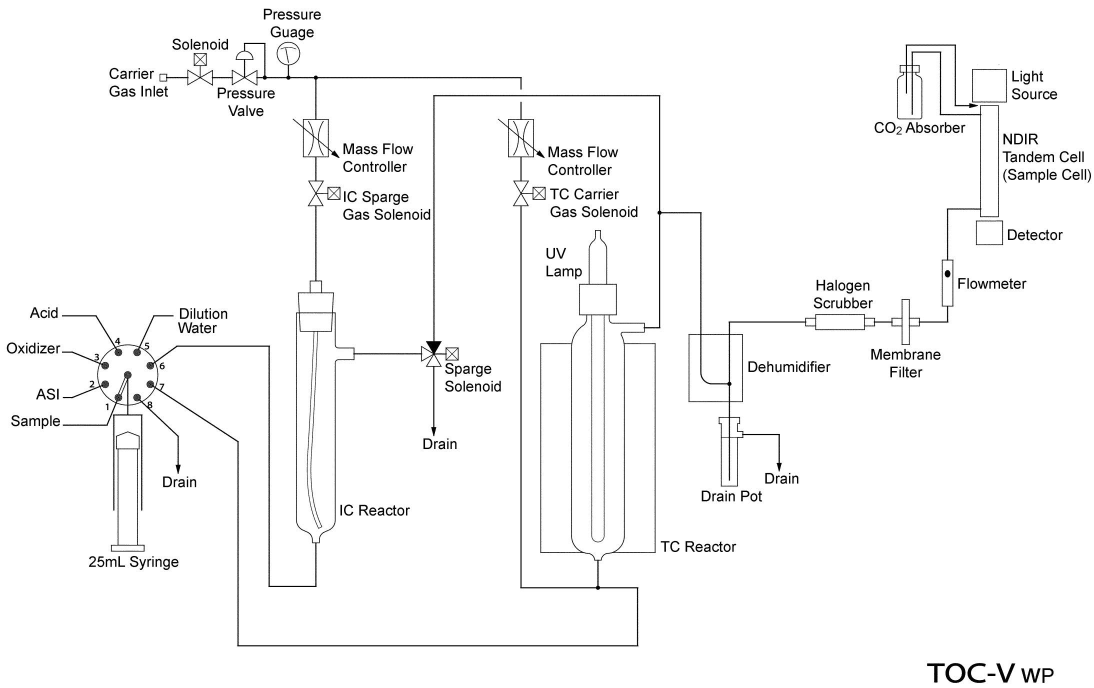


Figure 2.7 Flow Diagram (TOC-VWP)

2.2 ASI-V Autosampler (Option)

2.2.1 Front View

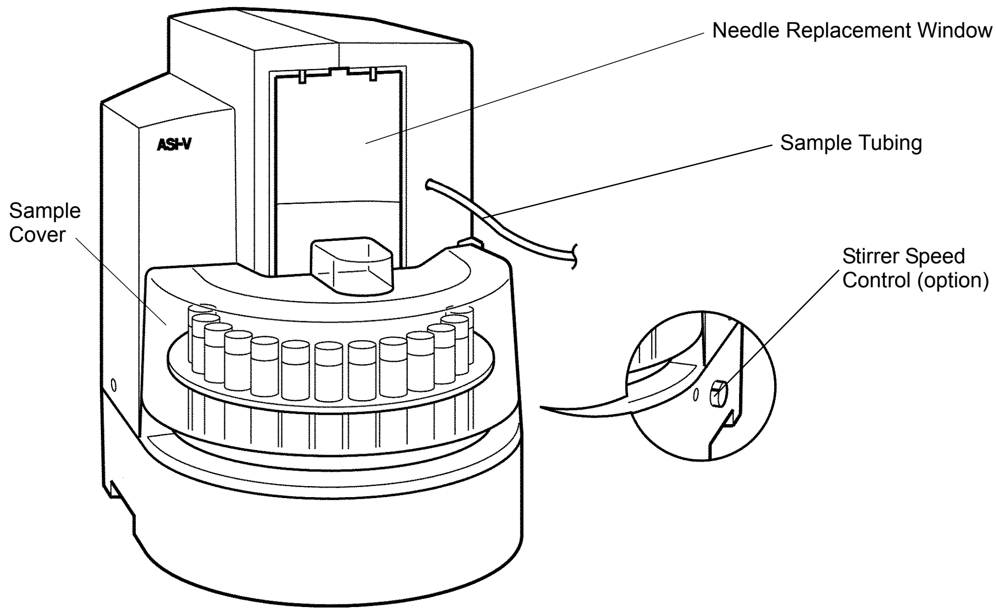


Figure 2.8 Front View

2.2.2 Rear View

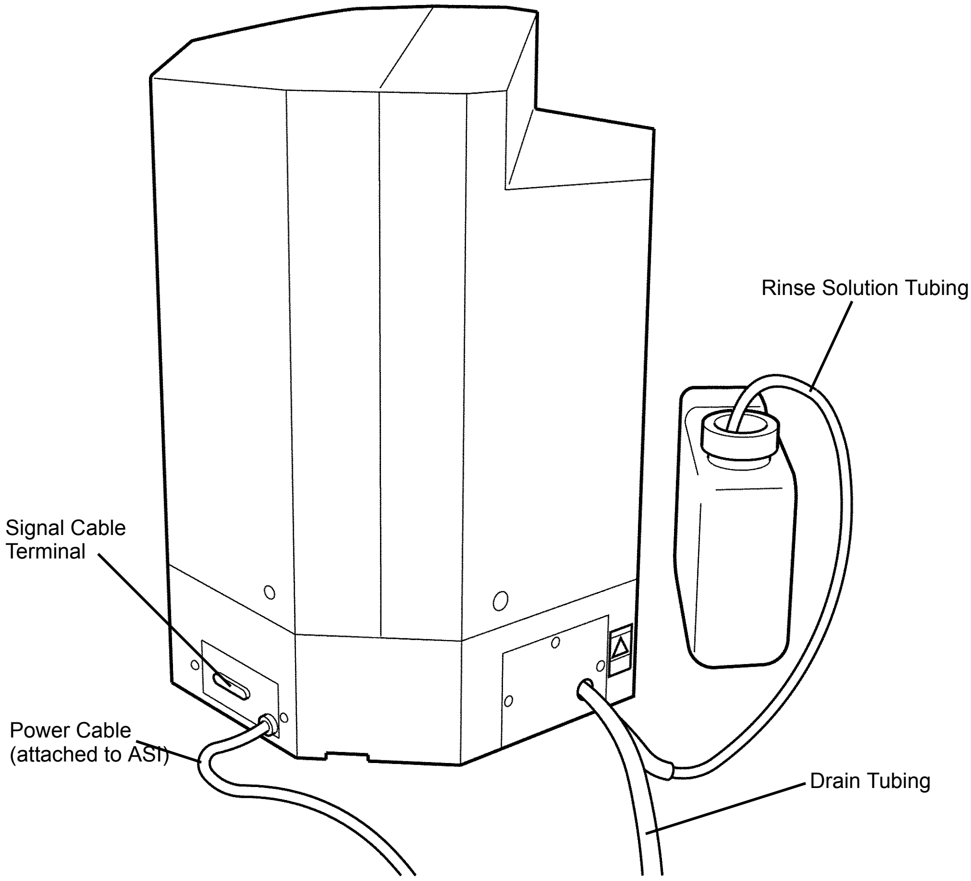


Figure 2.9 Rear View

2.3 OCT-1 8-Port Sampler (Option)

2.3.1 Front View

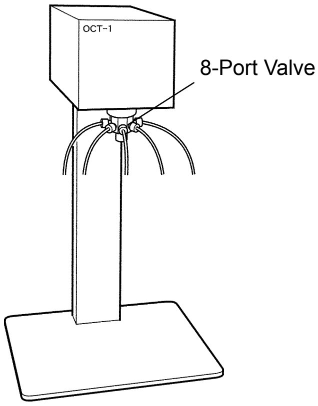


Figure 2.10 Front View

2.3.2 Rear View

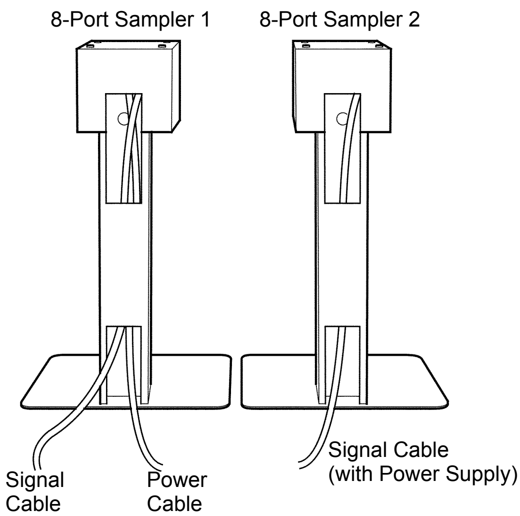


Figure 2.11 Rear View

3

Software Overview, Administration, and Setup

This chapter provides an overview of the TOC-Control V software and describes the Administration module and Instrument Setup Wizard.

3.1 Introduction to System Administration Functions

This section describes the various system administration functions in the TOC-Control V software that are available to the user. For a description of system administration functions that can be performed by the system administrator, refer to the TOC-V Administrator's Manual.

3.1.1 Screen Lock Function

If user must temporarily leave the PC during measurement, the screen can be locked to protect the data from another individual making improper or malicious operations in TOC-Control V.

This function can be used only if the User ID / Password item is checked in the Security dialog box.

To lock the screen, select Screen Lock in the Tools menu. The screen is locked and the Re-enter Password dialog box displayed. Once the screen is locked it can only be unlocked by entering the User ID and Password of the user who was logged on at the time the screen was locked.

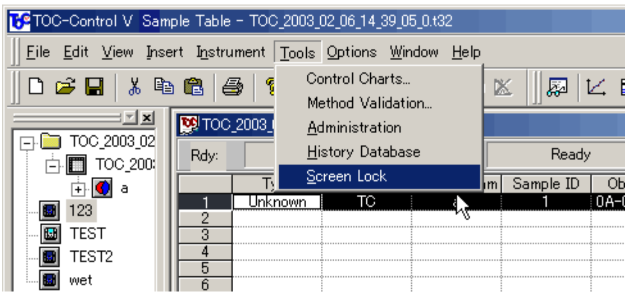


Figure 3.1 Screen Lock Command

Figure 3.2 Re-enter Password Dialog Box

3.1.2 Change Password

This function is used to change the password of a registered user.

Select Change Password in the TOC-Control V Administrator window to open the Change Password dialog box. This function can be used only if the User ID/Password item is checked in the Security dialog box. Refer to Section 3.2.4 “Security” in the Administrator’s Manual for more information.

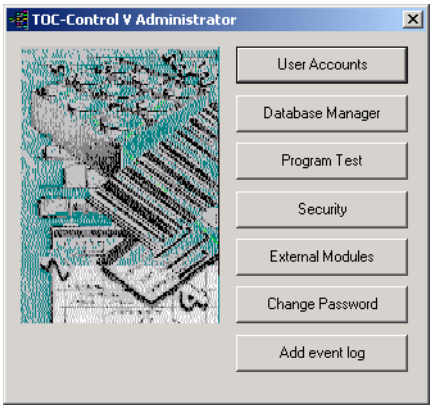


Figure 3.3 TOC-Control V Administrator Window

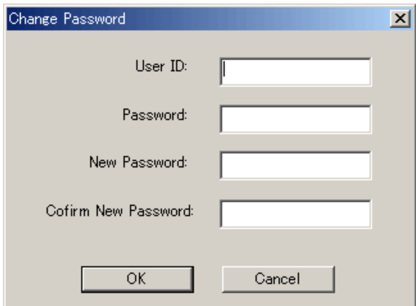


Figure 3.4 Change Password Dialog Box

3.1.3 Add Event Log

This function is used when a comment is to be included with the operation history. This function is useful for documenting items in the operation history, such as the replacement of catalyst, that cannot be recorded automatically through the software. Only users with access rights to Add Event Log can use this function.

This function can be used only if Audit Trail is checked in the Security dialog box. Refer to Section 3.2.4 “Security” in the Administrator’s Manual for more information.

When Add Event Log is selected in the TOC-Control V Administrator window, the Add Event Log dialog box is displayed.

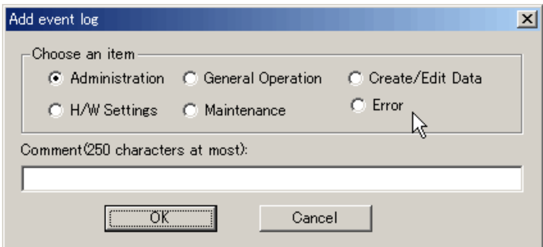


Figure 3.5 Add Event Dialog Box

Select one of the items in the Select Item array, and then enter a comment in the Comment text field. If the Comment text field is left blank, an error message is displayed, and the comment is not added to the Event Log.

3.2 Instrument Setup and System Properties

This section describes the Instrument Setup Wizard, which is used to configure new systems. This section also describes procedures for viewing the properties of and removing previously-configured systems.

The TOC-Control V Instrument Setup Wizard is used to enter properties for a new system. Access the Wizard by double-clicking the "New System" icon in the TOC-Control V main window.

Multiple instruments can be configured for the TOC-Control V software system by entering information into the series of Wizard screens.

TIP: *Use the Back and Next buttons to move among the various Instrument Setup Wizard windows.*

3.2.1 System Information Window

The System Information window of the Instrument Setup Wizard is used to enter basic system information.

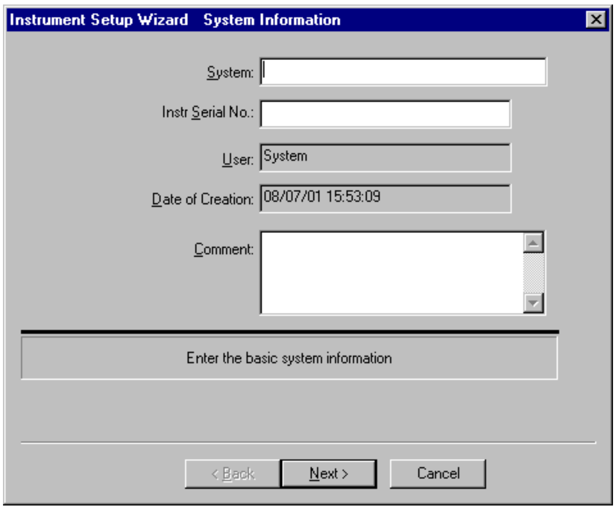


Figure 3.6 Instrument Setup Wizard: System Information Window

System

Enter a unique name for the system, using up to 32 characters. A name must be entered before continuing to the next Wizard page. If a system with the entered name already exists, an error message is displayed requesting entry of a unique name.

Serial Number

Enter the serial number of the TOC-V. The serial number of the unit is an 8-character number (No. ********) that is printed on the plate attached to the lower right side of the instrument.

User

The name of the current user is automatically entered into the User field and cannot be edited.

Date of Creation

The current date is automatically entered into the Date of Creation field and cannot be edited.

Comment

Enter additional system information in the Comment text box. The Comment field is optional and can contain up to 512 characters.

3.2.2 Options Window

The Options window of the Instrument Setup Wizard is used to specify the instrument hardware configuration and attached accessories.

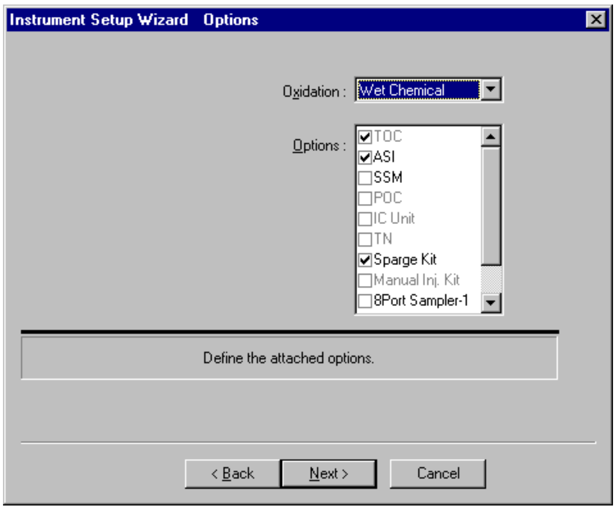


Figure 3.7 Instrument Setup Wizard: Options Window

Oxidation

Select "Wet Chemical" from the drop-down list to specify the oxidation method.

Options

Select the installed instrument components by clicking in the appropriate check boxes. When [Wet Chemical] is selected as the oxidation method, [ASI], [SSM], [Sparge Kit], [8Port Sampler-1], and [8Port Sampler-2] can be selected. The selected options determine which Instrument Setup Wizard screens are displayed.

Option	Description
TOC	Displays the TOC screen of the Instrument Setup Wizard. This option is always selected.
ASI	Displays the ASI screen of the Instrument Setup Wizard to allow autosampler parameters to be set. ASI cannot be selected if SSM or 8-Port Sampler 1or 2 are selected
Sparge Kit	Specifies that the Sparge Kit accessory is installed and that external sparging is desired. If the Sparge Kit is selected as an option, the "Needle" parameter is enabled on the ASI screen of the Instrument Setup Wizard.
8-Port Sampler 1	Select this option when one 8-port sampler accessory is installed. 8-Port Sampler 1 cannot be selected if the ASI or SSM are selected.
8-Port Sampler 2	Select this option when two 8-port sampler accessories are installed. 8-Port Sampler 2 can only be selected if 8-Port Sampler 1 is selected.
SSM	Select this option when the SSM (solid sample module) accessory is installed and necessary for measurement. the SSM cannot be selected if the ASI or 8-Port Sampler 1 or 2 is selected.

3.2.3 TOC Window

The TOC window of the Instrument Setup Wizard is used to set TOC instrument parameters.

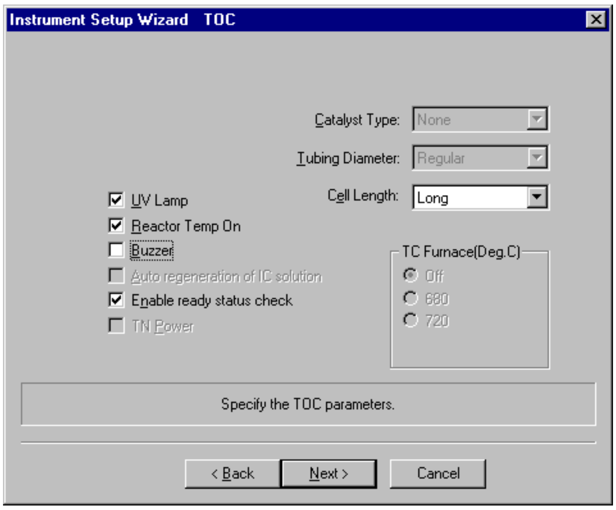


Figure 3.8 Instrument Setup Wizard: TOC Window

Cell Length

Select the length of the NDIR cell from the drop-down list. Available options:

- Long: for typical measurements.
- Short: for measurement of extremely high concentration samples.

UV Lamp

Turns on the UV lamp inside the TC reactor.

Check this box to perform TC or NPOC measurement.

Reactor Temp ON

Turns on the heater of the TC reactor.

Check this box to perform TC or NPOC measurement.

Buzzer

Enables the internal buzzer function. Select this option to allow the buzzer to sound during measurement.

Enable Ready Status Check

Select this option to enable the Ready Status Check function. When the function is enabled, the software always verifies that the instrument is in the Ready state prior to starting measurement. Enabling the Ready Status Check function is recommended. When the function is deselected, measurement can be performed when the instrument is not in the Ready state, an option that is mainly for use by service personnel.

3.2.4 ASI Window

The ASI window of the Instrument Setup Wizard is displayed if ASI was selected in the Options window of the Wizard. Use the ASI screen to set the autosampler parameters.

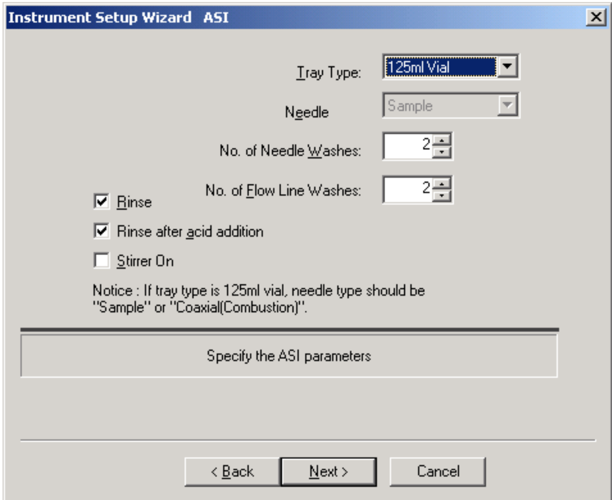


Figure 3.9 Instrument Setup Wizard: ASI Window

Tray Type

Select the ASI tray type from the drop-down list. Options are 40mL Vial and 125mL Vial.

Needle

Select the needle type from the drop-down list. Options are:

- Sample: for analysis using only a sample needle
- Sample+Sparge: for analysis using both a sample needle and a sparge needle

The Needle field is enabled only if Sparge Kit was selected in the Options window of the Wizard.

Note: The instrument does not automatically recognize the type of needle installed. Be sure to select the type of needle that is actually installed in the instrument.

No. of Needle Washes

Enter the number of times (1-10) for the outside of the needle to be washed after each sample using purified water from the ASI water bottle.

No. of Flow Line Washes

Enter the number of times (1-10) for the flow lines to be washed after all samples listed in the Sample Table have been measured. Purified water from the ASI water bottle is used to wash the flow lines.

Rinse

Select this option to rinse the sampling needle, syringe and flowlines between each sample with purified water from the ASI water bottle.

Rinse after acid addition

Select this option to rinse the sampling needle after each acid addition with purified water from the ASI water bottle.

3.2.5 SSM Window

The SSM window of the Instrument Setup Wizard is displayed if SSM was selected in the Options window of the Wizard. Use the SSM screen to set the SSM parameters. Refer to the SSM-5000A User Manual for details.

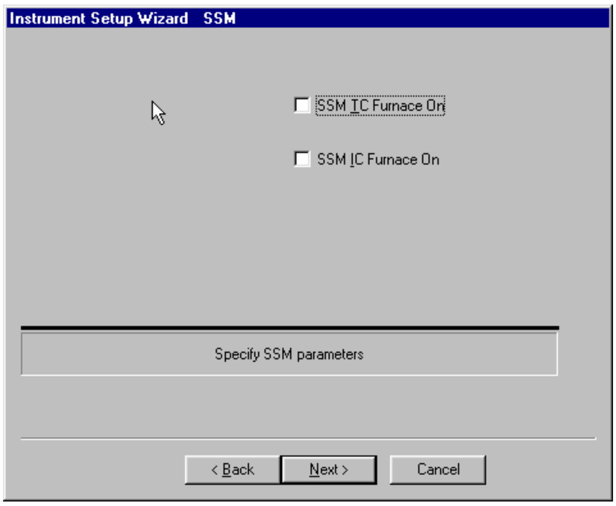


Figure 3.10 Instrument Setup Wizard: SSM Window

SSM TC Furnace On

Select this option for TC measurement.

SSM IC Furnace On

Select this option for IC measurement.

3.2.6 Communication Window

The Communication window of the Instrument Setup Wizard is used to set parameters for communication between the instrument and the PC.

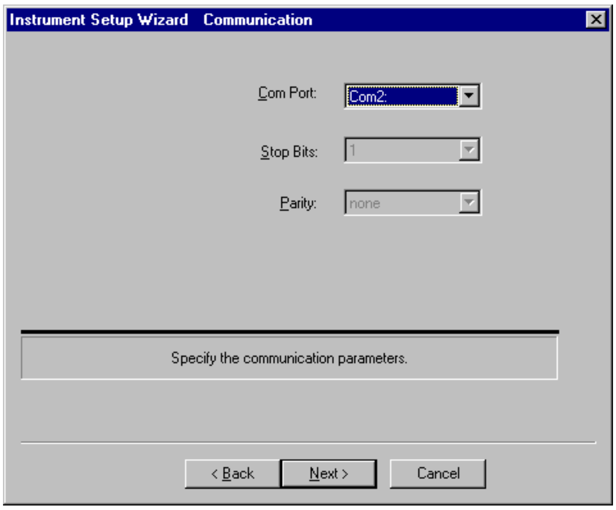


Figure 3.11 Instrument Setup Wizard: Communication Window

Com Port

Select the communication port from the drop-down list. Options include Com1, Com2, Com3, and Com4.

Stop Bits

The Stop Bits value is determined by the hardware and is set automatically based on the Com Port selection. This field is disabled and cannot be edited by the user.

Parity

The Parity value is determined by the hardware and is set automatically based on the Com Port selection. This field is disabled and cannot be edited by the user.

3.2.7 History Function Window

The History Function window of the Instrument Setup Wizard is used to enable the History Log. The History Log records all modifications to the system properties. In addition, the History Log can be set to require users to enter comments for each modification.

Note: Once the History Log is enabled, it cannot be disabled. All modifications to the system properties are tracked. If the mandatory comment option is selected, the user is required to enter a comment each time any system parameter is modified.

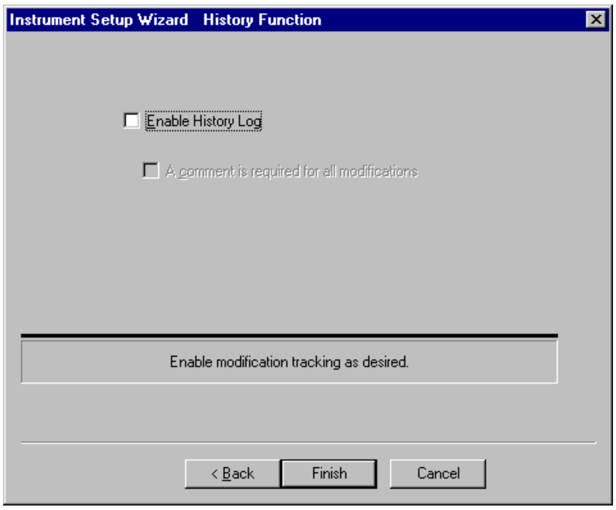


Figure 3.12 Instrument Setup Wizard: History Function Window

Enable History Log

Select this option to enable the software to monitor all modifications to the system properties. Once the History Log is enabled, the check box is disabled and the log cannot be turned off. The History Log is maintained on the History tab of the Instrument Properties dialog box. Refer to Section 3.2.8 "Instrument Properties Dialog Box" for more details. If the History Log is not required, it need not be enabled, as the software operates properly without it.

A Comment is Required for All Modifications

Select this option to require the user to comment on or obtain approval for all modifications. Once this option is selected, the check box is disabled and the option cannot be turned off. If the History Log is not required, it need not be enabled, as the software operates properly without it.

Finish Button

The History Function window is the final screen in the Instrument Setup Wizard. Make the appropriate selections, then click the Finish button. The new instrument is created and saved, and its icon appears in the TOC-Control V main window.

3.2.8 Instrument Properties Dialog Box

Instrument parameters can be viewed and modified using the Instrument Properties dialog box, shown below. Open the dialog box by double-clicking the instrument icon in the TOC-Control V main window.

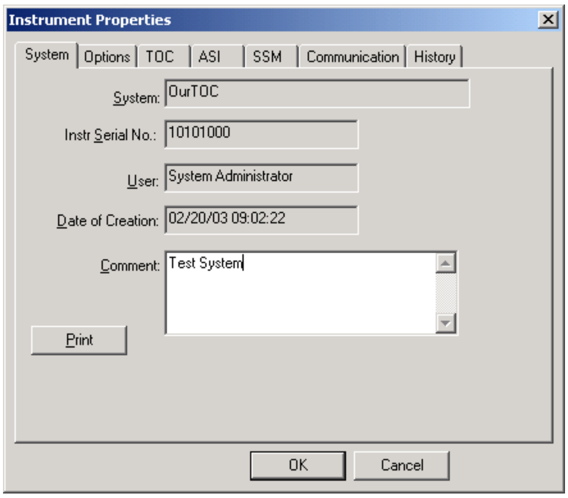


Figure 3.13 Instrument Properties Dialog Box

System Tab

The System tab displays the System Information window for the selected system and allows editing of the comments field.

Options Tab

The Options tab displays the oxidation method and instrument components that were selected in the Options window of the Instrument Setup Wizard. Information on this tab can be viewed but not changed.

TOC Tab

The TOC tab displays the TOC window. Some fields on the TOC tab can be edited. If the History Log was enabled during setup, edits to the TOC tab are tracked and listed in the History tab.

ASI Tab

The ASI tab displays the ASI window. Some fields on the ASI tab can be edited. If the History Log was enabled during setup, edits to the ASI tab are tracked and listed in the History tab.

Communication Tab

The Communication tab displays the Communication window. Some fields on the Communication tab can be edited. If the History Log was enabled during setup, edits to the Communication tab are tracked and listed in the History tab.

History Tab

The History tab displays the History Function window. If the History Log was not enabled during setup, this screen can be used to enable the History log.

If the History Log was enabled during setup, the History tab lists all changes made to the system parameters. The log lists modifications chronologically, with the most recent modification at the top. If the mandatory comment option was selected during setup, all modifications listed in the History tab window contain a comment describing the reason for the change. View or edit previously entered comments by selecting the modification from the list and clicking the Edit Comment button. If the History Log is not required, it need not be enabled, as the software operates properly without it.

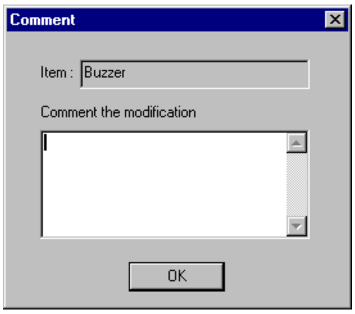


Figure 3.14 Comment Dialog Box

If the mandatory comment option was selected in the History Function window during instrument setup, the Comment dialog box (shown above) is displayed after any change is made in the Instrument Properties dialog box.

3.2.9 Removing a Configured System

Systems are removed from the TOC-Control V software using the main window. To remove a system, right-click the appropriate system icon and select Delete. Once a system is removed, it cannot be restored.

4

Operation

This chapter describes procedures for pre-analysis preparations, setting measurement parameters, starting up the instrument, generating calibration curves, and conducting sample analysis. This chapter also includes a tutorial that covers setup and measurement procedures.

4.1 Analysis Preparation

4.1.1 Installing the TC reactor

Installation Procedure

1. Loosen both of the TC reactor cover screws, and remove the cover.

2. As shown in Figure 4.1 "Connection of TC Reactor and Fitting", connect the joint fitting to the narrow end of the TC reactor.

   Note: Orient the joint fitting perpendicular to the TC reactor branch tube. When facing the branch tube. the blue tubing is connected to the right side of the fitting, and the white tubing to the left.

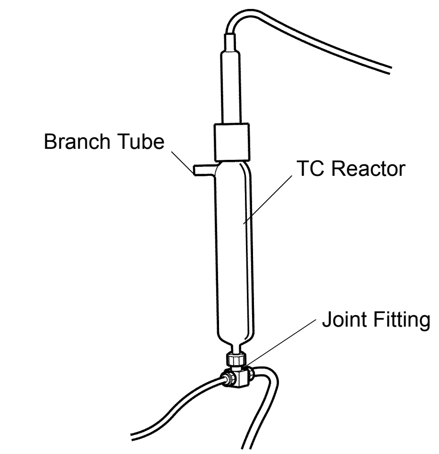


Figure 4.1 Connection of TC Reactor and Fitting

3. As shown in Figure 4.2 "Connection of TC Reactor and Viton Connector", connect the Viton connector to the TC reactor branch tube, and fasten it with the hose clamps.

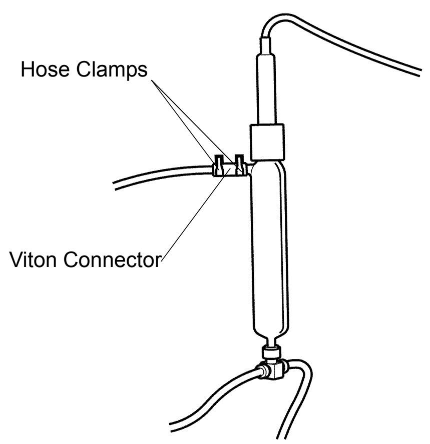


Figure 4.2 Connection of TC Reactor and Viton Connector

4. Remove the six (6) TC reactor heater insulator retaining pins, then open the two (2) heater retaining brackets to open it up.

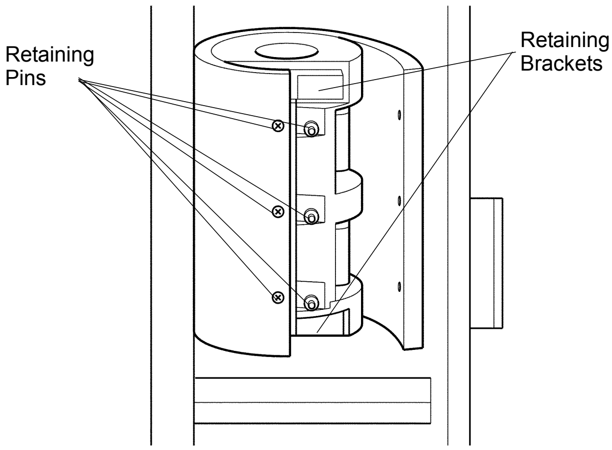


Figure 4.3 TC Reactor Heater

5. Roll the heat conducting sheet into a tube. With the opening of the sheet on the opposite side from the branch tube, slide the TC reactor into the tube and then slide the rolled sheet into the TC reactor heater.

6. Align the branch tube of the TC reactor with the notch on the back of the heater surface. Verify that the seam in the heat conducting sheet faces the front of the TC reactor heater, close the TC reactor heater, and close the retaining brackets.

   Note: If the inside of the TC reactor can be seen through the space in the heat conducting sheet, the TC reactor is correctly installed.

7. Open the top cover, and connect the UV lamp connector to the UV lamp power supply as indicated on the circuit board.

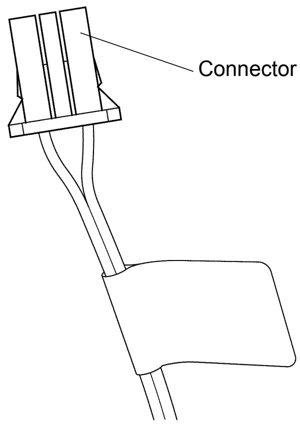


Figure 4.4 UV Lamp Cable Connector

8. Replace the cover on the TC reactor. Pass the tubing and wires through the slit in the TC reactor cover.

9. Close the top cover.

CAUTION

- Turn OFF the TC reactor heater and the UV lamp, and wait for the heater to cool to room temperature before performing any maintenance on the TC reactor. There is a danger of burn injury and electric shock.
- High voltage is applied to the UV lamp and UV lamp power supply. Due to the danger of electric shock, turn OFF the UV lamp before performing any maintenance on the TC reactor.
- The TC reactor is constructed of glass. To avoid injury, do not break the glass when connecting the TC reactor.
- When tightening the plastic nuts used on the connectors, do not use any tools. Tightening securely by hand is sufficient.
- Do not contaminate internal or external surfaces of the TC reactor or the inside of the connectors with organic substances such as grease or oil. This is critical when performing high sensitivity measurement.

4.1.2 Installing the IC Reactor

Installation Procedure

1. As shown in Figure 4.5 “Connection of IC Reactor and Fitting”, connect the joint fitting to the narrow end at the bottom of the IC reactor.

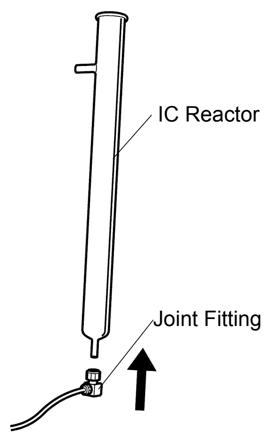


Figure 4.5 Connection of IC Reactor and Fitting

2. As shown in Figure 4.6 “Connection of IC Reactor and Viton Connector”, connect the Viton connector to the IC reactor branch tube, and fasten it with the hose clamps.

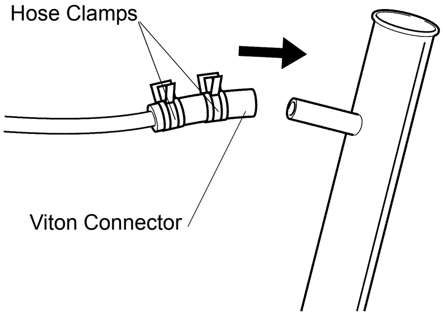


Figure 4.6 Connection of IC Reactor and Viton Connector

3. Insert the sparge tube into the IC reactor, and secure it by tightening the cap screw.

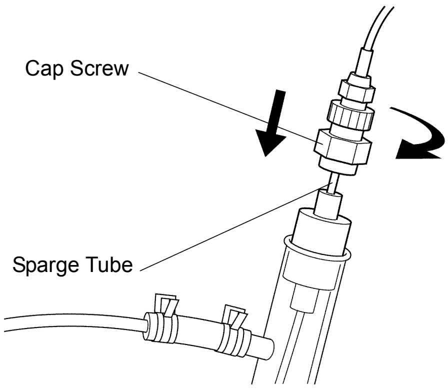


Figure 4.7 Inserting the Sparge Tube

4. Attach the IC reaction tube to the instrument using the mounting clamp.

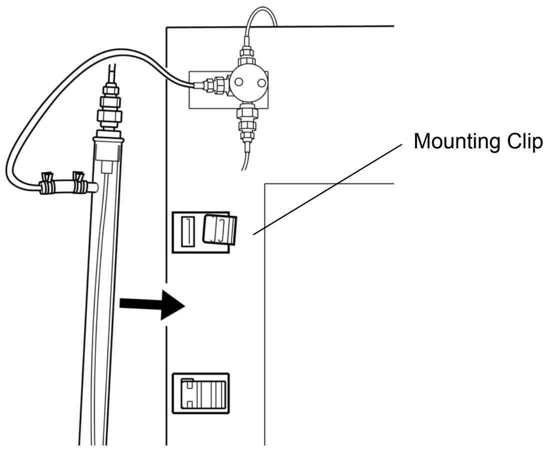


Figure 4.8 Attaching the IC Reaction Tube

CAUTION

- The IC reactor is constructed of glass. To avoid injury, do not break the glass when installing the IC reactor.
- When tightening the plastic nuts used on the connectors, do not use any tools. Tightening securely by hand is sufficient.
- Do not contaminate internal or external surfaces of the IC reactor or the inside of its connectors with organic substances such as grease or oil. This is critical when performing high sensitivity measurement.

4.1.3 Installing the Syringe

This section describes the procedure for attaching the syringe to the syringe pump assembly.

Depending on the instrument status, flow lines are connected to each of the 8 ports of the syringe pump valve. If the syringe is installed or removed without following the sequence described below, acid can be released from the syringe connection port. The sequence described below must be followed.

CAUTION

- Due to the potential for injury, keep hands away from the sample injector during operation of the syringe pump unit.
- The syringe barrel is made of glass and must be handled carefully to avoid breakage.

Syringe Replacement Procedure

1. Open the TOC-Control V Sample Table Editor and connect the instrument.
2. From the Instrument menu, select Maintenance>Change Syringe.

   The Syringe Change dialog box is displayed.

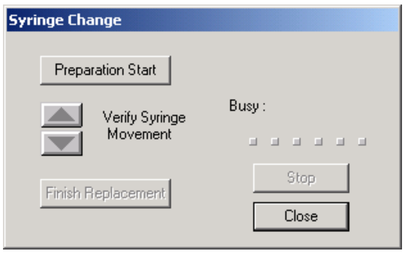


Figure 4.9 Syringe Change Dialog Box

3. Click the Preparation Start button to begin the procedure. A progress bar is active while the syringe moves to the position where it can be replaced.

4. When the progress bar begins blinking, remove the screw at the bottom of the syringe pump cover, and remove the cover.

5. Remove the syringe from the syringe connection port of the 8-port valve.

6. Attach the new syringe to the syringe connection port of the 8-port valve.

Note: Do not use tools when replacing the syringe; hand-tighten only. Over-tightening can deform the components of the 8-port valve and result in leaks.

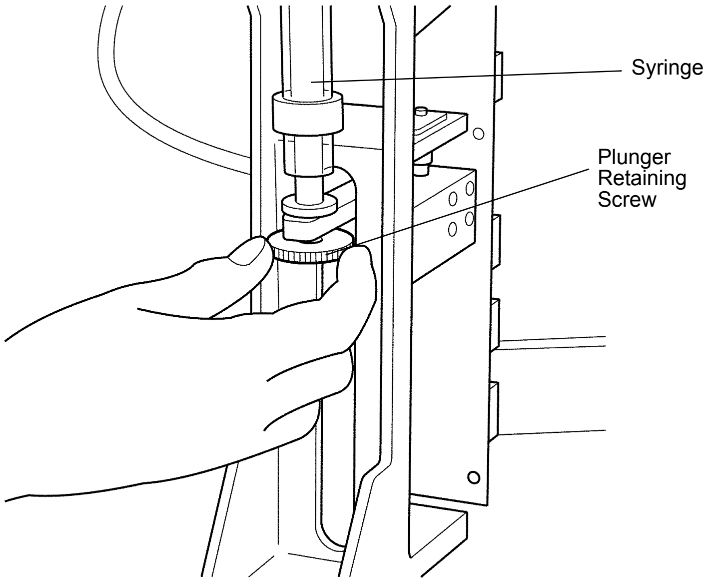


Figure 4.10 Installing the Syringe

7. Verify that the syringe moves smoothly between the upper and lower positions by clicking the directional arrows on the Syringe Change dialog box.

8. With the syringe in this position, hand-tighten the plunger retaining screw on the plunger holder.

9. Click the Finish Replacement button to move the syringe to its initial position.

10. Click the Close button.

Note: Once the syringe is mounted, perform the syringe pump zero point detection operation.

4.1.4 Syringe Pump Zero Point Detection

This section describes the procedure for performing automatic detection of the syringe pump zero point.

Note: Perform the zero point detection procedure whenever a new syringe is installed or the syringe is replaced.

Procedure

1. Open the TOC-Control V Sample Table Editor and connect the instrument.

2. From the Instrument menu, select Maintenance>Zero Point Detection.

   The Zero Point Detection dialog box is displayed.

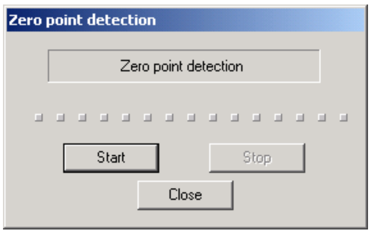


Figure 4.11 Zero Point Detection Dialog Box

3. Click the Start button.

   A progress bar is active while the syringe moves to the position where the zero point can be detected. When the progress bar stops blinking, the procedure is complete.

4. Click the Close button.

4.1.5 Water Supply to the Dehumidifier Drain Container

The drain container must be filled with purified water to prevent carrier gas release from the drain tubing.

Procedure

1. Remove the rubber cap from the drain container.
2. Remove the drain container from the installation clip.
3. Introduce purified water (deionized water is acceptable) into the dehumidifier drain container up to the level of the drain discharge tube on the side of the container.
4. Cap the drain container with the rubber cap.
5. Return the drain container to its position in the installation clip.
6. Verify that the dehumidifier drain tubing reaches to the bottom of drain container.

CAUTION

Do not bend drain tubing, as this may prevent drain discharge.

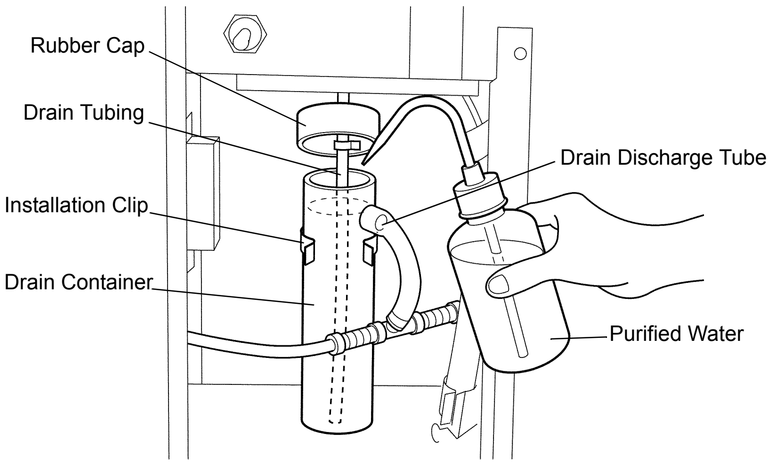


Figure 4.12 Filling the Dehumidifier Drain Container

4.1.6 Preparing the Persulfate Oxidizer Solution

To prepare the oxidizer, dissolve 60g of sodium persulfate and 15mL of phosphoric acid (85%) in purified water, and bring to a total volume of 500mL. This solution consists of 10.5% sodium persulfate and 3.8% phosphoric acid. It is not necessary to measure the reagents with great accuracy. A ±2% degree of accuracy is adequate. It is necessary, however, to keep the contaminant concentration in both reagents as low as possible. While reagent grade phosphoric acid should be used, it is not easy to find reagent grade sodium persulfate. Use sodium persulfate with as low a contaminant concentration as possible. Directly after preparing the oxidizer, blank values are high, resulting in an adverse affect on accuracy for high sensitivity analysis. It is therefore recommended that the persulfate preparation be allowed to stand in a cool, dark location for 24 hours before use. The persulfate solution is stable for approximately one month when stored in a cool, dark location.

Preparation Procedure

1. Prepare the persulfate solution by dissolving 60g of sodium persulfate and 15mL of phosphoric acid (85%) in purified water, and diluting to 500mL with purified water.
2. Wash the provided persulfate bottle with laboratory detergent, rinse well with tap water, and then rinse several times with purified water.
3. Rinse the persulfate bottle several times with small amounts of the persulfate solution, discarding the rinse volume after each rinse. Transfer the remaining persulfate solution to the bottle.
4. Place the persulfate bottle on the left side of the instrument.
5. Pass the tubing labeled PERSULFATE through the hole in the cap of the bottle.
6. Attach a cable tie to the tubing to prevent it from being pulled out of the cap.
7. Place the cap on the persulfate bottle.

   Note: Verify that the tip of the tube reaches all the way to the bottom of the bottle.

⚠ CAUTION

Sodium persulfate and phosphoric acid are corrosive chemical substances. Do not touch or spill these substances. Refer to Section 6.9 "Material Safety Data Sheets" for details.

4.1.7 Preparing Acid

To prepare the acid, mix 100mL of reagent grade phosphoric acid (85%) with purified water, and bring to a total volume of 500mL. It is not necessary to measure the reagent with great accuracy. A ±2% degree of accuracy is adequate. The phosphoric acid solution is stable for approximately one month when stored in a cool, dark location.

Preparation Procedure

1. Prepare the acid solution by mixing 100mL of phosphoric acid (85%) in purified water, and diluting to 500mL with purified water.

2. Wash the provided acid solution bottle with laboratory detergent, rinse well with tap water, and then rinse several times with purified water.

3. Rinse the acid solution bottle several times with small amounts of the acid solution, discarding the rinse volume after each rinse. Transfer the remaining acid solution to the bottle.

4. Place the acid solution bottle on the left side of the instrument.

5. Pass the tubing labeled H_3PO_4 through the hole in the cap of the bottle.

6. Attach a cable tie to the tubing to prevent it from being pulled out of the cap.

7. Place the cap on the acid bottle.

   Note: Verify that the tip of the tubing reaches all the way to the bottom of the bottle.

⚠ CAUTION

Phosphoric acid is a corrosive chemical substance. Do not touch or spill this substance. Refer to Section 6.9 “Material Safety Data Sheets” for details.

4.1.8 Preparation of Dilution Water

This section describes the preparation of water used for automatic sample dilution.

Procedure

1. Wash the provided dilution water bottle with laboratory glassware detergent, wash well with tap water, then rinse a number of times with purified water.
2. Fill the container with purified water to the 2 liter level.
3. Place the container on the right front side of the instrument.
4. Pass the dilution water intake tubing, labeled DILUTION, through the hole in the cap.
5. Attach a cable tie, about 200mm from the end of the tubing, to prevent the tubing from being pulled out of the cap.
6. Place the cap on the container.

   Note: Verify that the tip of the intake tubing nearly reaches the bottom of the container.

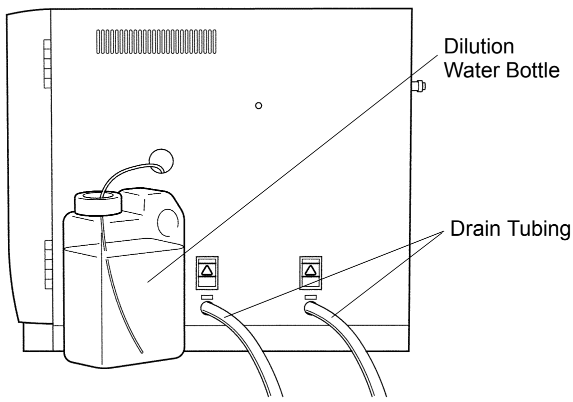


Figure 4.13 Dilution Water Bottle

Note: Purified Water

- Use the purest water available.
- To obtain good measurement reproducibility, it is recommended that the water in the dilution water bottle be replaced with fresh purified water every day.
- Refer to Section 4.1.11.1 “Preparation of Standard Solutions” for details on the degree of purity for “purified water.”

4.1.9 Installation of the CO_2 Absorber

A CO_2 absorber is used to remove CO_2 from the carrier gas that purges the optical system of the detector. The CO_2 absorber is installed using the following procedure.

Procedure

1. Cut the tips of the rigid tubes (2) in the lid of the CO_2 absorber with a knife.
2. Connect the flexible tubing that exits the rear panel of the instrument near the top as described below.

   Tubing labeled “L”: Connect to tube labeled “L” on CO_2 absorber.

   Tubing labeled “S”: Connect to tube labeled “S” on CO_2 absorber.

   Note: The flexible tubing should not be bent.
3. Place the CO_2 absorber in the holder behind the instrument.

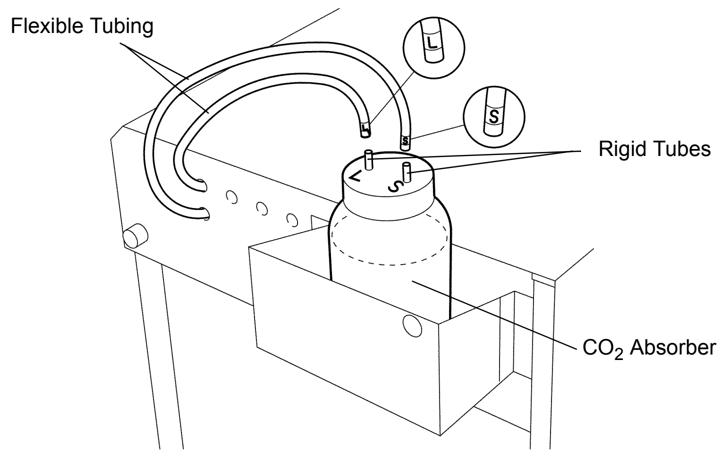


Figure 4.14 CO_2 Absorber

4.1.10 Adjusting Sparge Gas Flow Rate

The sparge gas flow rate should be adjusted before sparging is performed using the optional external sparge kit. The procedure for adjusting the sparge gas flow rate is described below.

Procedure

1. Open the TOC-Control V Sample Table Editor and connect the instrument.
2. From the Instrument menu, select Maintenance>Sparge Gas Valve.

   The sparge gas solenoid valve opens and sparge gas begins to flow.
3. Open the front door of the instrument.
4. Adjust the flow rate to about 100mL/min using the sparge gas flow rate adjustment knob.

   Note: The flow rate should be adjusted according to the size and shape of the sample container.
5. Close the front door of the instrument.

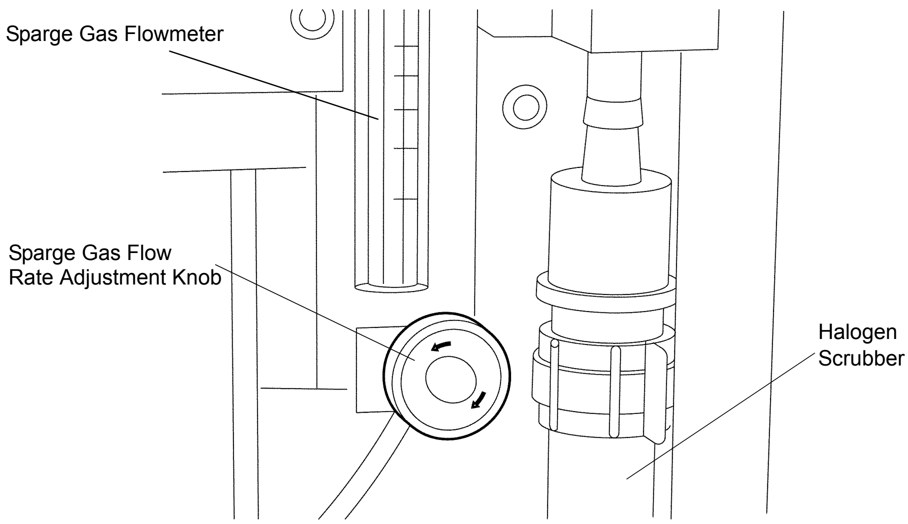


Figure 4.15 Sparge Gas Flow Rate Adjustment

4.1.11 Preparation and Storage of Standard Solutions

4.1.11.1 Preparation of Standard Solutions

Zero Water

Zero water refers to a standard solution with zero concentration of TC or IC, or the water used to prepare these standard solutions. Water that is in theory absolutely free of carbon (TC) is used as zero water. It is difficult, in practice, to obtain this type of water. Even ultra-pure water obtained using a high-performance membrane technique or water that has been repeatedly distilled has a TC content of about 10µg/L. This value is measured directly after manufacture, and increases when the water is stored in a container. Carbon dioxide is contained in the atmosphere at 300-500mg/L. The CO_2 content is higher in a room with many people, or where combustion equipment is used. This carbon dioxide dissolves in the water and is present as IC. The dissolved amount is generally about 0.2mg/L, but is dependent on the water temperature and carbon dioxide content of the air in contact with the water. Refer to Table 6.2 "CO_2 Content (ppm) in Distilled Water Equilibrated with Atmosphere Temperature (°C)". Instruments that prevent contact with air are not incorporated in water up-take, retention or storage during common distillation and purification of water. For this reason, IC is present in most distilled or purified water. There are cases where the TC content in distilled water commercially available in plastic or glass containers is 1mg/L.

Note: The purity of water required for zero water varies depending on the analysis range. There would be no problems using commercially available purified water when a 100mg/L sample is measured. The quality of the zero water should be considered along with the analysis range.

Preparation of TC Standard Solutions

1. Accurately weigh 2.125g of reagent grade potassium hydrogen phthalate previously dried at 105-120°C for about 1 hour and cooled in a desiccator.

2. Transfer to a 1L volumetric flask and dissolve in zero water.

3. Add zero water to the 1L mark, and stir the solution.

   The carbon concentration of the solution is equivalent to 1000mg/L and will be denoted as 1000mg C/L. This solution is retained as the standard stock solution. Here, the standard concentration of 1000mg/L is denoted as 1000mgC/L.

   Note: The standard stock solution may be prepared at other concentrations. For example, a 2000mg C/L solution may be used as the standard stock solution.

4. The standard stock solution is diluted with zero water to prepare standard solutions at the required concentrations.

   Note: The TC standard solution reagent need not be potassium hydrogen phthalate. Depending on the application, other substances are also acceptable (e.g., sucrose, etc.).

Preparation of IC Standard Solution

1. Accurately weigh 3.50g of reagent grade sodium bicarbonate (bottle labeled sodium hydrogen carbonate) previously dried for 2 hours in a silica gel desiccator, and 4.41g of sodium carbonate previously dried for 1 hour at 280-290°C and cooled in a desiccator.
2. Transfer the weighed materials to a 1L volumetric flask.
3. Add zero water to the 1L mark.
4. Stir well to mix.

   This solution is equivalent to 1000mg C/L and is referred to below as the “IC Standard Solution”.

4.1.11.2 Storage of Standard Solutions

The standard solutions undergo concentration changes when stored even for short periods, particularly the low-concentration solutions. Therefore, it is recommended that high concentration standard stock solutions (for example, 1000mgC/L) be stored in airtight containers in a cool, dark place. Glass bottles are suitable storage containers.

Storage Time

The limitation on storage of standard solutions is about 2 months for 1000mg C/L standard stock solutions, and about 1 week for diluted standard solutions (for example, 100mg C/L). These limitations are for sealed containers in cold storage.

Note: IC standard solution absorbs atmospheric carbon dioxide and undergoes concentration changes, so it is particularly important to store this solution in a sealed container.

Prepare Fresh Standard Solutions

- If reproducibility of analysis values is poor, or concentration values fluctuate.
- If contaminants, even small amounts of dust, are present in the standard solution.

4.1.12 Sample Preparation

4.1.12.1 High Sensitivity Analysis

High sensitivity analysis refers to measurement of trace amounts (commonly 0.5mg/L or less) of TOC in samples such as ultra-pure water. The precautions described in the sections below should be followed when conducting trace analysis on the order of 10-100µg/L.

Precautions for High Sensitivity Analysis

Measure Samples Immediately

Samples containing trace carbon, such as ultra-pure water, should be measured immediately after collection. If analysis cannot be performed immediately, store the sample in a dark location without freezing at 0-10°C, and perform analysis as soon as possible.

To collect the water, introduce water into a well-washed container, leaving no headspace, and seal the container.

Use Glass Sample Containers

Glass containers are optimal for use as sample containers. 500mL - 1L commercially available glass screw-lid jars with Teflon seals (generally referred to as heat-resistant screw-top jars or hermetic jars) are appropriate for use as water collection and transport containers.

Note: Exercise caution when using plastic containers, as the elution of TOC components may occur.

Use NPOC for Analysis of Ultra-pure Water

Use the NPOC method for analysis of ultra-pure water samples. TOC determination using the difference between TC and IC is prone to large analysis errors for the following reasons:

- The TOC value is significantly affected since a large portion of TC often comprises IC. Errors in TC analysis and IC analysis are additive.
- Carbon dioxide is absorbed from the atmosphere during sample collection and analysis. During the elapsed time between TC analysis and IC analysis, the sample absorbs more carbon from the atmosphere, causing the IC result to be higher than the IC content in the TC result. Therefore, subtracting the elevated IC result from the TC yields a biased TOC value.
- The TOC value can also be greatly influenced if standard solutions are not prepared and measured carefully. Errors are compounded since two calibration curves must be generated for TC and IC.
- In NPOC analysis, POC (purgeable organic carbon) may be volatilized from the sample during sparging for IC removal. This is not a problem since the ultra-pure water contains practically no POC.

Calibration Curve Generation and Sample Analysis

Minimize contamination of the sample due to the addition of oxidation acid by limiting the amount of acid added to obtain a final sample pH of 4 or less. Determine the appropriate volume of acid to be added beforehand, by carrying out trial oxidation using a graduated cylinder, etc.

If the volume of acid added is less than 100µL, analytical reproducibility decreases due to fluctuation in the volume of acid added. Establish the absolute volume to be added to be at least 100µL, though the addition volume is actually set using a percentage of the volume of sample injected.

A 2-point calibration curve that passes through the origin is used for sample measurement. The case below describes the generation of a 2-point calibration curve shifted to pass through the origin. The concentrations used are 0µg/L and 400µg/L.

Cautions in Calibration Curve Generation and Sample Measurement Using High Sensitivity Analysis

The case below describes the generation of a 2-point calibration curve shifted through the origin. The calibration standard concentrations are 0µg/L and 400µg/L.

Preparation of Standard Solutions

A 2-point calibration curve using 0µg/L and 400µg/L is generated. The calibration curve is shifted through the origin to correct for the TC contained in purified water used for preparation of the standard solutions.

The 0µg/L and 400µg/L TC standard solutions should be prepared at the same time using the same purified water. Wash well, two 250mL volumetric flasks with purified water (preferably water containing low TC concentration). Carefully introduce 1mL of 100mg/L TC standard stock solution (for a 400µg/L solution) into one of the volumetric flasks. Distilled water is introduced up to the graduation line in both volumetric flasks.

Standard preparation procedures should be performed as quickly as possible, using caution to avoid inclusion of external contaminants. If a water purification apparatus is the source of purified water, allow the water to flow for a few minutes prior to use, as the initial water will have a higher TC concentration. Alternate the flasks a number of times while filling them with water to equalize, as much as possible, the TC concentration of the purified water.

Use fresh purified water when filling the volumetric flask to the graduation line. Do not use rinse bottle water that has been standing for a long period of time because the TC concentration continually increases with time.

Instrument Preparation

The blank peaks present at the start of high sensitivity analysis will become progressively smaller and gradually stabilize. As a result, calibration curves generated too soon after the start of analysis may be inaccurate because they contain data collected during unstable conditions. To prevent this problem, perform 5 or more measurements of purified water containing 2-3 drops of 1M HCl per 100mL of water prior to analyzing the calibration standards. Use the same measurement parameters (injection volume, sparge time) that will be used for the calibration standards and samples.

Calibration Curve Analyses

Calibration curves are generated using the following two methods.

1) Using standard solution in a 250mL volumetric flask

   Insert the sample tubing directly into the volumetric flask until it reaches the bottom of the flask. Perform analyses quickly. Four to five repeated analyses can be made with almost no effects from the absorption of atmospheric carbon dioxide. Cover the opening of the volumetric flask with sealing film or paraffin to prevent contamination. Use this method when the IC value of the purified water used for standard solution preparation (specifically, 0µg/L standard solution) is about 50µg/L or less.

2) Sparging the standard solution

   To sparge in the sample injection syringe, set the acid addition ratio to 0.5%, and the sparge time to 2 minutes. To sparge using the optional external sparge kit, transfer about 60mL of the standard solution to a clean sample container, add 1-2 drops of 2M hydrochloric acid, sparge for 10 minutes and analyze.

   Cover the sample container with paraffin or sealing film to prevent contamination. A glass container with an external diameter of 24-25mm and a height of 200-250mm is appropriate. Test tubes, stopper-equipped test tubes and chromatography tubes of suitable size are available commercially. If the outer diameter of the container is too large, the air-to-water contact efficiency is decreased, requiring a longer sparge time. Ensuring that the tip of the sparge tube is near the bottom of the container will improve the sparging efficiency.

Reference: *Refer to Section 4.3.2 "Tutorial" and Section 4.4.2.2 "Calibration Curve" for detailed descriptions of generating calibration curves using the TOC-Control V software.*

Sample Analysis

Use the standard curve generated in the previous section and measure the NPOC of the sample. Measure the sample using the same method described in method 2) above for measuring standard solutions. If the values gradually decrease with each repeated analysis using the optional external sparge kit, extend the sparge time to ensure sufficient sparging. The shape of the sample container, sample volume, amount of sparge gas and IC content of the sample all have a bearing on the required sparge time. Determine the appropriate sparge time in accordance with these factors.

4.1.12.2 Pretreatment for IC Removal (Sparging)

Errors for TC and IC analyses are additive. For samples that have a comparatively high IC content relative to the TOC content, the TOC analysis error is large if it is determined by the difference between TC and IC results. Samples of this type include samples from the natural environment such as rivers, lakes, swamps, oceans and ground water, or samples from water treatment facilities.

Using the difference between TC and IC to determine TOC is not appropriate for purified water or ultra-pure water samples. This is because the CO_2 in air dissolves in the sample and is measured as IC; this concentration tends to vary.

In such cases, the sample is pretreated with aeration or sparging before analysis to remove the IC. The sample is then subjected to TC analysis, immediately followed by TOC analysis using the NPOC method.

Sparge Sequence

There are 2 methods available for performing sparging with this instrument.

1) Sparging within the IC reactor for a set time, followed by measurement

   Refer to Section 4.3.2 “Tutorial” for details on the measurement method.

2) Independent sparging of sample using the optional external sparge kit

   Transfer the sample to a clean sample container, acidify the sample and then perform sparging. A glass container with an external diameter of 24-25mm and a height of 200-250mm is appropriate, however, to generate a calibration curve with a full scale of 50µg/L or less, 20-70mL of standard solution are required (for repeat measurement), requiring a larger container. There are test tubes, stopper-equipped test tubes and chromatography tubes of suitable size available commercially. If the outer diameter or the container is too large, the sparge gas-to-water contact efficiency is decreased, requiring a longer sparge time.

Sparge Procedure

1. Start delivery of the sparge gas using the procedure described for adjusting the gas flow.

Reference: *Refer to Section 4.1.10 "Adjusting Sparge Gas Flow Rate" for details.*

2. Add concentrated phosphoric acid (85%) which has been diluted by a factor of 5 to acidify the sample to a pH of 2-3.

3. Insert the sparge tubing into the sample.

   To improve the sparging efficiency, the tip of the sparge tubing should be as close to the bottom of the sample container as possible.

By this method, sparge gas can be used independently. While the first sample is being measured, the next sample can be sparged, thereby improving analysis efficiency.

About Sparge Time

In nearly all cases, IC is adequately removed by sparging within the IC reactor for 3 minutes. When the concentration of IC is high, for example 10mg/L of IC, set the sparge time to 4-5 minutes to remove 99+% of the IC.

Conversely, when the concentration of IC is extremely small, as in the case of purified water samples, a sparge time of less than 3 minutes should be sufficient to achieve adequate IC removal.

If the optional external sparge kit is used to perform sparging outside the instrument, or if sparging is performed inside the vial placed in the autosampler, processing conditions must be investigated as mentioned above.

4.1.12.3 Persulfate Addition Volume

The persulfate solution is added automatically in TC and NPOC measurement. The volume of persulfate to be added is set in the Conditions setting screen. The default addition volume is 1.5mL, which usually provides adequate oxidation for analysis.

If the peak shape broadens and the TC reactor temperature, carrier gas flow and other instrument conditions are normal, the situation may be improved by increasing the volume of persulfate. This helps sharpen the peak shape if the sample inadvertently contains non-dissolved organic substances. Increasing the amount of persulfate beyond the default values should only be used in such instances. Changing the volume of persulfate added to the sample when it is not necessary chemically results only in using more persulfate. If it is determined that all of the organic substances in the sample are easily decomposed, the volume of persulfate may be reduced based on examination of the factors (recovery rate examination). If the volume added is smaller than 100µL, reproducibility is adversely affected due to fluctuation of the volume of persulfate added. Therefore, set the addition amount so that the actual volume added is at least 100µL.

4.1.12.4 Acid Addition Volume

The acid solution is added automatically in IC and NPOC measurement. The volume of acid to be added is set in the Conditions setting screen. The default volume is 3% of the total volume of sample processed in the IC reactor. This usually provides adequate acidification but may be insufficient depending on the sample characteristics. If the IC measurement peaks broaden under normal carrier gas flow and other instrument conditions, or if IC removal is insufficient in NPOC measurement, increasing the volume of acid so that the final pH is 2-3 may be effective. The volume added should be determined by trial testing beforehand. Conversely, when performing high sensitivity analysis, the volume added can be reduced to suppress the background from the acid. See Section 4.1.12.1 "High Sensitivity Analysis" for more details.

Note: In analyses where IC exceeds 5mg/L, the sample injection volume displayed on the screen is less than 3mL. The sample volume is diluted to 3mL with the addition of dilution water to the sample in the IC reactor. The volume of acid is calculated based on this total volume.

The total volume used to perform sparging for NPOC measurement is about 5mL at least, depending on the number of measurement repetitions and whether or not auto dilution of the sample is conducted. The volume of acid added is calculated as a percentage of the total sample volume.

4.1.12.5 Measurement of Samples Containing Suspended Solids

In the TOC-VWP, persulfate reagent is added to the sample, which is then subjected to ultraviolet irradiation and heat to oxidize the organic component of the sample. The recovery rate varies for samples that contain non-dissolved organic substances or suspended particles. This is most obvious when the non-dissolved organic substances are inadvertently decomposed or if substances are difficult to decompose causing them to be oxidized non-uniformly.

Therefore, it is important to filter sample suspensions and to analyze only the filtrate. When the sample contains sedimenting suspended particles, set the sample aside and allow the solids to completely settle out, then analyze the supernatant liquid.

If samples are analyzed with the suspended particles present, wash the TC reactor (Section 5.2.5 "Washing the TC Reactor") and IC reactor (Section 5.2.6 "Washing the IC Reactor") frequently to prevent buildup of residual suspended solids in the flow line, which could cause carryover or blockage.

Heavy solids that settle quickly, such as soil and sand, remain at the bottom of the syringe and can scratch the rotor of the 8-port valve. Separate out such sedimentation before analysis.

Countermeasures

Perform one of the following processing procedures before analyzing samples that contain suspended particles.

1) TOC measurement of sample supernatant

Set the sample suspension aside and allow the particles to settle to the bottom of the sample container. Use only the supernatant liquid as the sample. Depending on the sedimentation characteristics of the suspended particles, the soluble TOC (DOC: Dissolved Organic Carbon) is analyzed in this method.

2) TOC measurement of filtrate

The filter paper or membrane filter may contain TOC that could contaminate the sample. Heat the glass filter paper before use. Then use water or the sample solution to wash away any TOC in the filter.

Filter the sample through glass fiber paper or a membrane filter to remove the suspended particles. The dissolved TOC in the filtrate is analyzed.

4.1.12.6 Handling Samples Containing Acids, Bases or Salts

When samples contain halogens, various adverse effects such as reduction in the recovery rate and poor peak shape are evident in TC and NPOC analysis. The degree to which these effects are manifested depends on the absolute amount of the halogen injected into the TC reactor.

The table below gives the maximum concentration limits at which the halogen (e.g., chloride ion) does not produce the above-mentioned effects.

Table 4.1 Halogen Concentration Maximum Limit (Example: Chloride Ion)

Calibration Curve Concentration	Injection Volume (Automatically set value)	Halogen Concentration Maximum Limit (Chloride ion)
1mg/L	3mL	500mg/L
50mg/L	0.5mL	3000mg/L

The effects of the halogen are relatively large with respect to the small peaks when measuring concentrations up to 1mg/L. If 50mg/L of chloride ion is present in a 10µg/mL sample, measurement is not possible.

When halogen is present in samples having high TOC concentrations, dilute the sample to reduce the adverse effects of the halogen. If the calibration curve concentration is set to 70mg/L or greater, dilution is performed automatically in the TOC analyzer (2-50 times, depending on the set value).

If sample dilution is not an option due to low TOC concentration, the injection volume might be reduced by using a larger calibration curve concentration setting. Measurement reproducibility may be decreased when a smaller injection volume is used.

4.1.13 Sample Preparation for Autosampler Measurement

4.1.13.1 Vial Types

There are 2 types of autosampler vials. Since the TOC-VWP is most often used for high sensitivity measurement or measurement of low concentration samples, use the 40mL and 125m vials because they conserve sample and reduce contamination of the sample from the inner wall of the vial. The 125mL vial is recommended for analyzing samples with extremely low concentrations on the order of 100µg/L.

Use the sample volumes indicated in the following table for the corresponding vial type. Using volumes less than those listed below may result in an insufficient amount of sample for analysis

Table 4.2 Vial Types

Vial	Typical Sample Volume[1]	Sample Volume for NPOC Analysis[2]
40mL	40mL	35mL
125mL	125mL	105mL

[1] Sample volume when vial is filled to the bottom of vial cap.
[2] Sample volume when vial is filled to 1cm from bottom of vial cap.

In NPOC analysis, the sample volume is decreased to reduce contact with the sample cap during sparging. Sparging cannot be performed in the vial when using 125 mL vials.

Note: Vials to be used for the first time should be washed with laboratory detergent and then rinsed well with purified water. Alternatively, certified precleaned vials can be purchased.

Maximum Number of Measurements by Vial Type

When using the 40mL and 125mL vials and the volume of sample indicated above, the relationships between the full-scale values of the calibration curve and the maximum possible number of measurements are indicated in the following tables. These are based on use of a single wash (default).

Table 4.3 40mL Vials

Calibration Curve Full Scale	Maximum Number of Measurements		
	NPOC Analysis	TC Analysis	TC-IC Analysis
25µg/L	1	1	_
50µg/L	2	2	1
100µg/L	3	4	2
200µg/L - 5mg/L	5	5	2
10mg/L	5	6	3
20mg/L	7	8	4
50mg/L - 3500mg/L	9 or more	10 or more	5 or more

Table 4.4 125mL Vials

Calibration Curve Full Scale	Maximum Number of Measurements		
	NPOC Analysis	TC Analysis	TC-IC Analysis
25µg/L	4	4	2
50µg/L	7	8	4
100µg/L	11	13	6
200µg/L - 3500mg/L	16 or more	18 or more	9 or more

4.1.13.2 Filling Vials with Sample

Under normal operation conditions, open vials may be used for analyzing samples with the autosampler. In the following situations, however, the vials must be sealed:

1) sparging with the autosampler for NPOC analysis
2) measuring acidic samples
3) IC analysis
4) POC analysis
5) low-concentration samples (as a guideline, less than 10mg/L)
6) alkaline samples (pH 7 or higher)
7) samples that contain volatile organic hydrocarbon components
8) a large number of samples to be measured
9) standard solutions

In situations (1) and (2) above, corrosion of the instrument may occur due to spattering of sample, which contains acid. If the samples are left in the autosampler following measurement, there is danger that the autosampler may be subjected to corrosion due to the volatilization of acid.

In situations (3) through (9), analysis values are affected by contamination of absorbed CO_2 from the atmosphere or volatilization of the TOC component in the sample.

Methods of Sealing Vials

There are two acceptable methods of sealing the sample vials.

1) Using the caps provided with the vials.

Note: Use caps only after washing them with water or laboratory cleaning solution, and then rinsing well with purified water.

2) Using a paraffin-based sealing film marketed under names such as Parafilm.

Using Caps

125mL vials

Caps and seals are provided with the vials.

1. Place a seal over the vial opening.

   Note: Because the seals are perforated during injection, discard the seals after use.

2. Place the vial cap over the seal.

For 40mL vials

Seal-equipped caps are provided with the vials.

1. Using a seal-equipped cap provides an adequate seal for the vial.

   Note: Because the seals are perforated during injection, discard the seal-equipped cap after use.

Using Paraffin-based Sealing Films

Procedure

1. Cut the sealing film into approximately 25mm x 25mm (1inch x 1inch) squares.

2. Place a sealing film square over the opening of the vial, and stretch it to seal it to the edges of the vials.

3. Press the edges down so that they wrap around the outside of the vial.

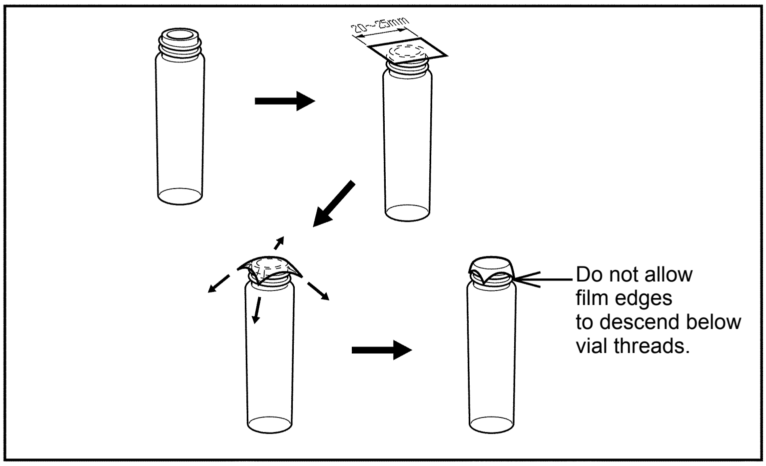


Figure 4.16 Sealing the Vial

4.1.13.3 Placing Vials in the Vial Rack

Remove the vial rack from the ASI-V before placing the sample vials in it. There are three different types of vial racks, each accommodating a different vial size.

The ASI-V permits random access of vials in the vial rack. The vials are measured according to the sequence set in the “ASI Schedule Settings” screen, thus analysis can start from any position number in the vial rack and occur in a non-sequential vial order.

TIP: *When samples are to be sparged using the optional external sparge kit, analysis can be performed more efficiently if the vials are placed in sequential rack-positions without gaps, so the vial adjacent to the vial being measured can be sparged.*

Installing the Vial Rack

Install the rack using the following procedure.

1. Place the vial rack containing vials on the ASI-V turntable.

2. Place the cover over the turntable.

   Note: During analysis, the turntable must be covered. The ASI-V will not operate if the turntable cover is not detected.

4.1.13.4 High Sensitivity Measurement using the Autosampler

When using the autosampler to analyze pure and ultra-pure water samples having carbon concentrations on the order of a few hundred µg/L, the following high sensitivity measurement precautions must be taken. Refer to Section 4.1.12.1 “High Sensitivity Analysis” for details.

- NPOC analysis is recommended because TOC (TC-IC) analysis tends to have a greater variation in measured values than NPOC.

  IC often accounts for a significant portion of the TC. Errors in the two values are additive using TOC (TC-IC) analysis. This significantly influences TOC analysis.

- For analysis of calibration curves, fill well-washed vials with standard solutions to about 5-10mm from the top and seal immediately.

  Use a cap that has been rinsed with purified water before use.

  When placing the vial in the rack, handle the vial carefully to prevent the standard solution contacting the cap.

  Low-concentration standard solutions must be prepared each time they are used.

- The automatic IC removal process of acidification and sparging for NPOC analysis can be performed inside the vial of the autosampler or in the TOC-VWP unit. The optional external sparge kit is required when performed inside the vial. Automatic IC removal should be performed in the TOC-VWP for sample concentrations up to 500µg/L for the following reasons.
    - There is a risk that a slight amount of IC may be re-dissolved into the sample during measurement when IC removal is performed inside the vial.
    - There is a risk of sample contamination when the needle pierces the seal during automatic acid addition in the vial.
- To stabilize the peaks, perform 4 - 5 injections using the same analysis parameters before performing the calibration curve analysis. For example, to generate a 400µg/L calibration curve, set 400µg/L as the 1st standard solution concentration, and enter 1 as the vial number. Place a 0µg/L solution in vial position 1. The objective is to inject water using the same injection volume used for the calibration curve. This stabilizes peaks and thoroughly washes the sample needle and sparge needle with purified water before generating the calibration curve.

  In the 2nd row of the settings screen and using the same calibration curve number as above, set the analysis parameters for 0µg/L and 400µg/L. Place the standard solutions in the rack after the stabilizing injections are complete.
- Acid addition for NPOC analysis can be performed automatically using the autosampler. It is best to perform manual addition of phosphoric acid for NPOC analysis on the order of 10µg/L. This is because, automatic acid addition pierces a hole in the seal, allowing CO_2 contamination from the air.
- If the number of needle washes is set to “0” in the “Instrument Settings - ASI” dialog box, the needle is not washed.

  Even if ultra-pure water is used for analysis on the order of 100µg/L, the concentration increases in the wash water during the time it is exposed. Therefore, better results are obtained in this situation if the needle is not washed.
- Do not use the analysis value acquired from the first sample vial. In sample analysis, particularly analysis on the order of 10µg/L, the first sample measured may yield high results.
- When using the optional external sparge kit, perform sparging using a sparge gas flow rate 100mL/min.

  As a guideline, use a sparge time of 5-10min for 40mL vials. Increase the gas flow rate or sparge time if sparging is insufficient.

4.1.13.5 Acid Addition in NPOC Analysis

Automatic addition of acid is possible using the autosampler when the optional external sparge kit is installed. Using the ASI-V, acidification and sparging can be performed on samples during NPOC analysis. To perform acid addition using the autosampler, first enter the acid addition amount in the “Measurement Parameters” screen.

Acid is drawn from the acid bottle into the syringe, and the set amount of acid is added from the sample needle into each of the vials that have been designated for acid addition. The phosphoric acid should be diluted 5 times. The acid addition ratio (ratio of acid with respect to sample) is generally set to about 3%. A setting of about 1% is used for purified water analysis.

The phosphoric acid concentration or amount added should be adjusted in accordance with the sample pH or buffering strength to bring the sample pH to 2-3. Before analysis, verify the pH using pH paper. The pH need not be checked each time if the same type of sample is routinely measured.

⚠ CAUTION

Phosphoric acid is a hazardous chemical. Exercise caution when handling to prevent contact and spills. Refer to Section 6.9 “Material Safety Data Sheets” for details.

If the sample pH does not reach between pH 2-3, increase the acid addition ratio or the acid concentration. Increasing the acid addition ratio results in dilution of the sample. If the acid amount is too small, the acid droplet on the tip of the needle may not become large enough to drop from the needle. Visually observe the acid droplet to confirm that it is actually added to the sample.

If [Rinse after Acid Addition] is selected in the “Instrument Settings - ASI” dialog box, and a value is entered for the number of washes, the needle is rinsed after every acid addition. To save time during the analysis sequence, washing of the needle is not required when there is very little variation in NPOC concentration among the samples.

4.1.13.6 Sparging in NPOC Analysis

By using the optional external sparge kit, the next sample vial in the autosampler tray can be sparged while the first sample is being analyzed. External sparging is not available with the 125mL vials.

If the additional sparge needle is used for the 40mL vials, the next vial to be measured can be sparged while the vial in the sampling position is being measured. Sparging is not performed again when the previously sparged vial is moved to the sampling position. This does not affect the mg/L level NPOC analysis.

For NPOC measurement of samples with concentrations in the order of several hundreds µg/L, perform the IC removal process in the TOC-VWP. Refer to Section 4.1.13.4 “High Sensitivity Measurement using the Autosampler” for details.

Verify that the sparge gas flow is not so great as to cause sample to splatter onto the seal of the vial cap.

Test the completeness of the IC removal by performing IC measurement on the sample remaining after NPOC measurement.

4.1.14 8-Port Sampler Measurement

4.1.14.1 Type of Sampler Container

There are no restrictions on the type of sample containers that can be used with the 8-port sampler. It is only necessary that the containers be placed so the sample tubing from the various ports can be inserted directly into the containers.

Note: Wash all sample containers with an appropriate laboratory detergent, and rinse them thoroughly with water before using them for the first time.

4.1.14.2 Loading the Sample

Although it is possible to perform measurement of a sample using the 8-port sampler while the container is open, in the following situations, it is necessary to seal the sample container.

1) When performing IC measurement
2) When performing POC measurement
3) When performing measurement of low concentration samples (e.g., 10 mg/L)
4) When performing measurement of alkali samples of pH 7 or higher
5) When performing measurement of samples containing volatile organic carbon compounds
6) When the number of samples is so great as to require a long measurement time
7) When performing measurement of standard solutions

Note: If the sample container openings are not sealed in the situations listed above, measurement values may be influenced by such factors as absorption of atmospheric CO_2, contamination with carbon containing substances, and evaporation of TOC constituents from the samples.

Sealing the Sample Containers

Sample containers may be sealed using commercial sealing films such as Parafilm. It is also possible to pierce the lid of the container and insert the sample tubing into the container through this opening.

Note: In either case, wash the container with a laboratory detergent, and rinse it thoroughly with water before use.

Sealing Procedure Using Parafilm

1. Cut a piece of Parafilm to size according to the size of the sample vessel opening.

2. Insert the tip of the sampling tube into the vessel, cover the opening with the prepared Parafilm, and seal the opening of the vessel, stretching the Parafilm over the edges. Be sure that a very small gap in the seal is formed to prevent creation of negative pressure inside the vessel.

3. Press the overlapping portions of the Parafilm around the neck of the opening.

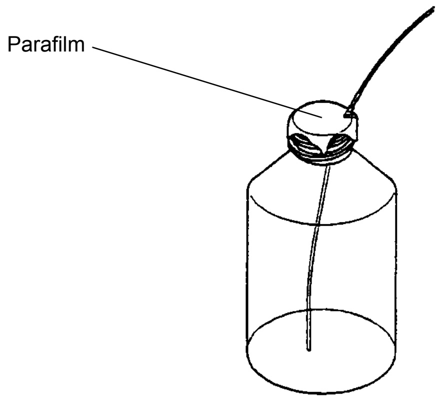


Figure 4.17 Sealing the Container

4.1.14.3 High Sensitivity Using the 8-Port Sampler

If samples such as purified water or ultra-pure water, having a TOC concentration of 100ppb or less, are to be measured using the 8-Port Sampler, it is necessary to consider the following precautions associated with high sensitivity analysis. Refer to Section 4.1.13.4 "High Sensitivity Measurement using the Autosampler" for more information.

1) NPOC analysis is recommended because TOC (TC-IC) analysis tends to have a greater variation in measured values than NPOC. This is because IC often accounts for a significant portion of the TC, and the errors in the two values are additive using TOC (TC-IC) analysis. This affect significantly influences TOC analysis.

2) For calibration measurement of standard solutions, fill well-washed containers with standard solutions to about 5-10 mm from the top of the container, and immediately seal each container. When placing the container below the 8-port sampler, handle it carefully to avoid the standard solution contacting the sealing surface. Low-concentration standard solutions must be prepared each time they are used.

3) To ensure that the peaks are stable for generating the calibration curve, perform 4-5 injections using the same analysis parameters prior to performing the calibration curve analysis. For example, to generate a 400ppb calibration curve, set 400ppb as the 1st standard solution concentration, and enter 1 as the container number. Actually place a 0ppb solution in the container for port No. 1. The objective at this point is not to generate a calibration curve, but to inject water using the same injection volume that will be used to generate the calibration curve. This not only stabilizes the instrument but also thoroughly washes the sampling needle with purified water.

   In the 2nd row of the settings screen and using the same calibration curve number as above, set the analysis parameters for 0ppb and 400ppb. When sample measurement starts, the calibration curve to be used will be created with the correct standard solutions.

4.2 Setting General Measurement Parameters

4.2.1 TOC Parameter Settings

The following parameters must be entered to perform analysis using the PC-controlled TOC-V analyzer: The items available for editing change depending on the options that are installed.

- UV Lamp
- Reactor Temp On
- Buzzer
- Enable ready status check

Setting Procedure

1. In the TOC-Control V main window, double-click the icon for the desired instrument.

The Instrument Properties dialog box is displayed.

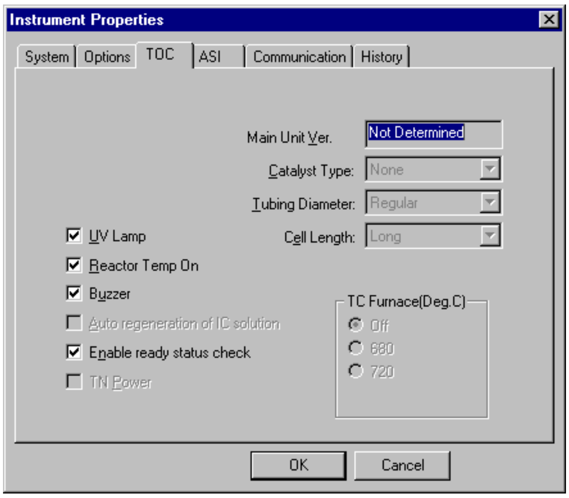


Figure 4.18 Instrument Properties Dialog Box: TOC Tab

2. Click the TOC tab.

The Main Unit Version field displays the instrument version number; this field cannot be changed by the user.

3. Enter the parameters as described below:

UV Lamp

Turns on the UV lamp inside the TC reactor. Place a check mark here to turn on the UV lamp.

Reactor Temp On

Turns on the TC reactor heater. Place a check mark here to turn on the heater.

Buzzer

Enables the internal buzzer function. Select this option to allow the buzzer to sound during measurement.

Enable ready status check

Enables the Ready Status Check function. When the function is selected, the software always verifies that the instrument is in the Ready state prior to starting measurement. Selecting the Ready Status Check function is recommended. When the function is deselected, measurement can be performed when the instrument is not in the Ready state, an option that is mainly for use by service personnel.

4.2.2 ASI Parameter Settings

The following parameters must be set when using the ASI-V:

- Tray type
- Needle
- No. of Needle Washes
- No. of Flow Line Washes
- Rinse
- Rinse after acid addition

Procedure

1. In the TOC-Control V main window, double-click the icon for the desired instrument.

The Instrument Properties dialog box is displayed.

2. Click the ASI tab.

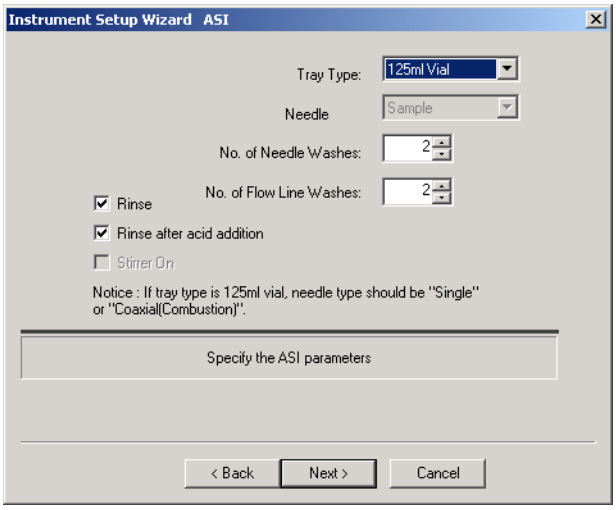


Figure 4.19 Instrument Properties Dialog Box: ASI Tab

3. Set the following parameters:

Tray Type

Specifies the size of the vials used in the ASI rack. Select the vial size from the drop-down list. The options are 40mL Vial and 125mL Vial.

Needle

Specifies the type of needle used for analysis. Select the needle type from the drop-down list. Options include:

- Sample: for analysis using only a sample needle. Sparging for NPOC measurement is performed inside the TOC-V unit.
- Sample+Sparge: for analysis using both a sample needle and a sparge needle. Sparging for NPOC analysis is performed inside the needle when both the when both the sampling and sparge needles are used with the external sparge kit.

Note: When using the 125mL vials, only a single needle can be used. Select the Sample needle option.

The instrument does not automatically recognize the type of needle installed. Be sure to select the type of needle that is actually installed on the instrument.

No. of Needle Washes

Specifies the number of times the needle is rinsed after sample injection. This operation rinses the outside of the needle using water drawn from the ASI water bottle. This function is not used for analysis of purified water and tap water samples.

Enter a number ranging from 0 to 10 in the No. of Needle Washes field.

No. of Flow Line Washes

Specifies the number of times the flow lines are rinsed after all samples listed in the Sample Table have been measured. This operation rinses the flow line from the sample needle to the injection tubing using water drawn from the ASI water bottle. If the automatic acid addition option has been selected, the flow lines are rinsed the specified number of times following acid addition to each sample; rinsing after acid addition removes acid that remains in the sample needle or flow lines. This function is not used for analysis of purified water and tap water samples.

Enter a number ranging from 0 to 10 in the No. of Flow Line Washes field.

Rinse

Enables automatic rinsing of the sample needle and sparge needle between each sample using water from the ASI water bottle.

- Rinsing between each sample prevents carryover from one sample to the next. Rinsing is not necessary if all samples contain similar carbon concentrations.
- Rinsing is not recommended when analyzing ultra-pure water samples with expected concentrations of less than a few hundred µg/L. The concentration of carbon in the rinse water may affect the sample results. Rinsing with ultra-pure water does not reduce this effect, as the concentration of carbon in the rinse water increases with exposure to the atmosphere.

Rinse after acid addition

Enables automatic rinsing of the sample needle and flow lines after each acid addition.

Note: Rinsing the needle after each acid addition is not necessary if all samples contain similar NPOC concentrations. Eliminating the rinsing operation saves time during the analysis sequence.

4.2.3 Setting Default Measurement Parameters

The Default Measurement Parameters window is used to enter the default values for sample measurement parameters. The selections made in the Default Measurement Parameters window are displayed as the default values when setting calibration curve and sample measurement parameters.

Procedure

1. From the Options menu in the Sample Table Editor, select Default Measurement Parameters.

The Default Measurement Parameters window is displayed.

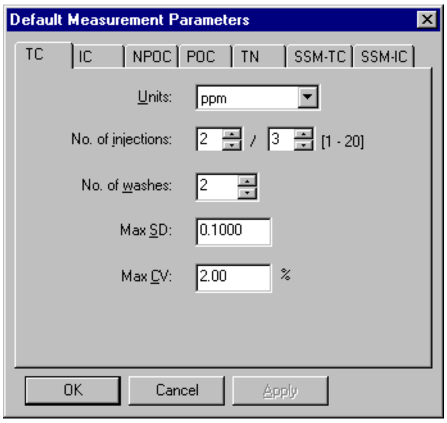


Figure 4.20 Default Measurement Parameters: TC Tab

2. Set the parameters on each of the applicable tabs.

- Units: Select the default concentration units for the measurement results.
- No. of injections: Enter the default minimum/maximum number of injections.
- No. of washes: Enter the default number of times the syringe is washed with sample prior to the first injection.
- Max SD: Enter the default maximum standard deviation that is acceptable for repeat measurements. If this value is met, no additional injections are required. However, if both this value and the Max CV value are exceeded, the samples are automatically reinjected up to the maximum number of times specified in the No. of Injections field. The Max SD field is disabled if the Number of Injections is 1 or if the minimum is equal to the maximum. The default value for Max SD is 0.1.
- Max CV: Enter the default maximum coefficient of variation that is acceptable for repeat measurements. If this value is met, no additional injections are required. However, if both this value and the Max SD value are exceeded, the samples are automatically reinjected up to the maximum number of times specified in the No. of Injections field. The Max CV field is disabled if the

Number of Injections is 1 or if the minimum is equal to the maximum. The default value for Max CV is 2%.

Note: The "maximum" No. of Injections field refers to the maximum number of repeat injections to be performed when the automatic additional measurement function is enabled. Additional measurements are performed when both the Max SD and Max CV settings are exceeded. When both of these values are exceeded for the set number of sample injections, the additional measurement function is automatically activated and the sample is reinjected.

4.2.4 Maintenance History Settings

Enter limits for the following parameters to display a warning message when the limits are exceeded:

- Dilution water (mL): Records the volume of dilution water used.
- Total Acid Vol (mL): Refers to the total volume of acid used.
- Syringe Stroke (no.): Refers to the number of syringe operations.
- UV Lamp (hour): Refers to the total UV lamp operation time.
- UV Oxid Vol (µL): Refers to the total volume of oxidizer used.
- ASI Rinse Pump (hour): Refers to time of ASI rinse pump tubing use. The recommended warning value is 300 hours.

Procedure

1. From the Instrument menu in the Sample Table Editor window, select Maintenance>History.

   The Maintenance History dialog box is displayed.

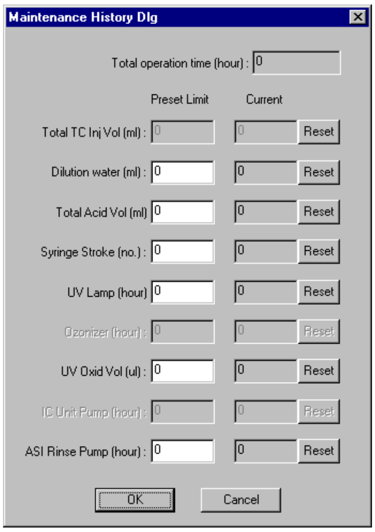


Figure 4.21 Maintenance History Dialog Box

2. Enter warning limits by placing the cursor in the appropriate fields and entering numerical values.

3. Click the OK button.

TIP: *Measurement is interrupted if the dilution water or acid reservoirs run dry during analysis. Prevent measurement interruption by setting the dilution water and total acid volume limits to values slightly less than the remaining volume of each respective liquid. A warning message is generated before each liquid is completely consumed.*

TIP: *Click the Reset button to the right of a parameter to clear its current value. Total Operation Time records the total time the instrument has been in operation and cannot be reset.*

4.3 Analysis

4.3.1 Starting Up the Instrument

4.3.1.1 Turning the Power ON

To perform analysis, turn on the power to the instrument as follows.

Procedure

1. Press the power button on the bottom right of the front panel of the instrument.

TIP: *Power Button positions:* I ON
O OFF

Note: Press the power button once more to turn power to the instrument off.

4.3.1.2 Carrier Gas Pressure

Set the TOC-V Carrier Gas Pressure as Follows.

1. Ultra high purity nitrogen gas is used as the carrier gas for this instrument. Set the supply pressure of the carrier gas to 300kPa (44PSI) using the pressure regulator at the carrier gas supply source (cylinder).

Note: Ensure that the supply pressure of the nitrogen gas is greater than 300kPa (44PSI) and does not exceed 600kPa (87PSI).

2. Open the front door of the instrument, and adjust the carrier gas pressure at the instrument to 200kPa (29PSI) using the carrier gas pressure regulation knob.

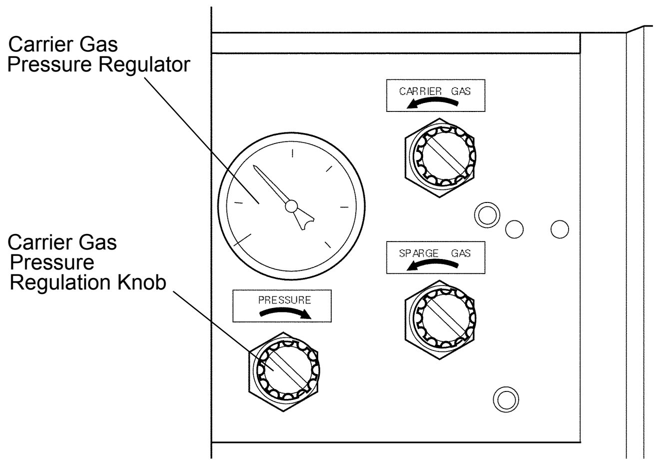


Figure 4.22 Setting the Carrier Gas Pressure

4.3.1.3 Carrier Gas Flow Rate

Set the flow rate of the TOC-V carrier gas as follows.

Procedure

1. Open the front door of the instrument.
2. Turn the carrier gas flow rate adjustment knob. Adjust the flow rate to 200mL/min as indicated on the flow rate gauge.

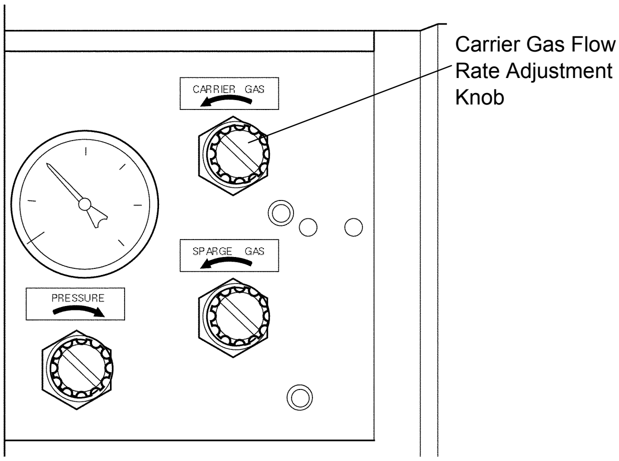


Figure 4.23 Setting Carrier Gas Flow Rate

Note: Do not change the carrier gas flow rate during analysis. The peak area changes with respect to flow rate in a nearly inverse proportional relationship and may cause errors in analysis precision.

4.3.1.4 Setting Sparge Gas Flow Rate

This sections describes the procedure for setting the flow rate of the sparge gas supplied to the TOC-V IC reactor.

Procedure

1. Open the front door of the instrument.
2. Select Maintenance>Adjust Sparg Flow Rate (Wet.Chem.)

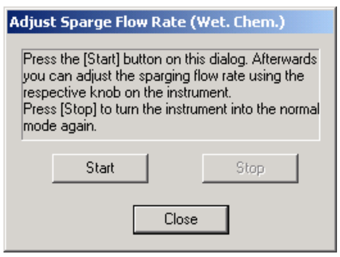


Figure 4.24 Adjust Sparge Flow Rate (Wet. Chem.) Dialog Box

3. Click the Start button.

4. Turn the sparge gas flow rate adjustment knob so that the flow meter indicates a flow rate of 200mL/min.

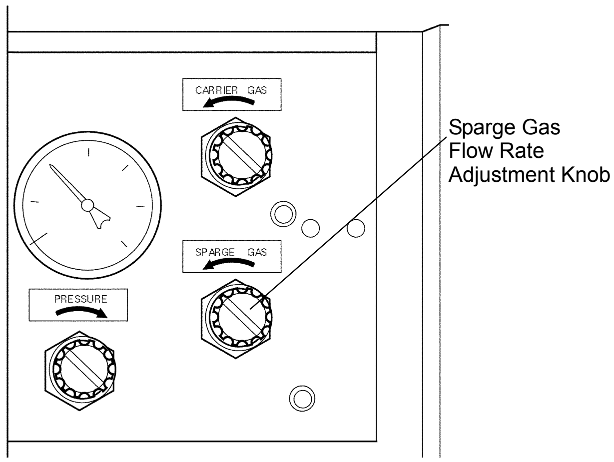


Figure 4.25 Adjusting the Sparge Gas Flow Rate

5. After making the adjustment, click the Stop button.

6. Click the Close button.

4.3.1.5 TC Reactor Settings

This section describes the procedure for setting parameters for the TC reactor. These parameters include the controls for the UV lamp and heater (Reactor Temperature On).

Procedure

1. In the TOC-Control V main menu, click the appropriate system.

   The Instrument Settings dialog box is displayed.

2. Click the TOC tab.

3. Place check marks next to UV Lamp and Reactor Temperature On.

4. Click OK.

   The display returns to the TOC-Control V menu.

4.3.2 Tutorial

This tutorial provides step-by-step instructions on the use of the TOC-Control V software. The tutorial assumes that the hardware and software have been installed in the manner described in Chapters 2 and 3, and that users have a configured system that includes the TOC-VWP and ASI.

Note: If your system does not include the ASI unit, adjust the tutorial procedures as appropriate.

In the tutorial, a three-point TC calibration curve and one unknown sample are measured. Purified water and the following calibration standards are needed for the tutorial:

- A calibration standard of 0mgC/L potassium hydrogen phthalate.
- A calibration standard of 5mgC/L potassium hydrogen phthalate.
- A calibration standard of 10mgC/L potassium hydrogen phthalate.

Reference: *Prepare calibration standards as described in Section 4.1.11.1 "Preparation of Standard Solutions".*

The following topics are covered in the tutorial:

1) Configuring the Instrument
2) Connecting the Instrument
3) Creating a Sample Table
4) Setting Up the Calibration Standard Runs
5) Setting Up an Unknown Sample Run
6) Performing Measurement
7) Evaluating the Results
8) Printing a Report

(Topic 1) Configuring the Instrument

Procedure

1. Start the TOC-Control V software by double clicking the TOC-Control V icon or by selecting it from the Start>Programs list.

The TOC-Control V main window is displayed.

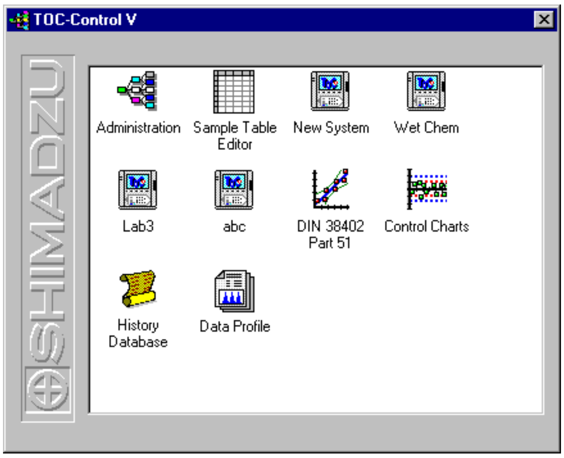


Figure 4.26 TOC-Control V Main Window

2. The instrument configuration must be specified in the software before the instrument can be connected to the PC for the first time. Only users with administrative privileges can configure the instrument. If the system has already been configured, proceed to Step 2 of the tutorial.

3. From the TOC-Control V main window, double-click the New System icon.

The User dialog box is displayed.

4. Enter your user name and password in the dialog box shown below.

Figure 4.27 User Dialog Box

5. Click the OK button. The Instrument Setup Wizard opens.

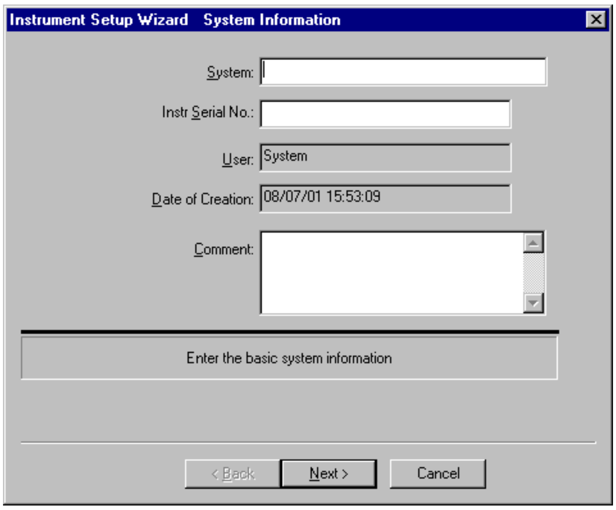


Figure 4.28 Instrument Setup Wizard: System Information Window

6. In the System Information window of the Instrument Setup Wizard, enter the System Name and Instrument Serial Number. Click the Next button.

   The Options window of the Instrument Setup Wizard is displayed.

Reference: *Refer to Section 3.2.1 "System Information Window" for details on the System Name and Instrument Serial Number.*

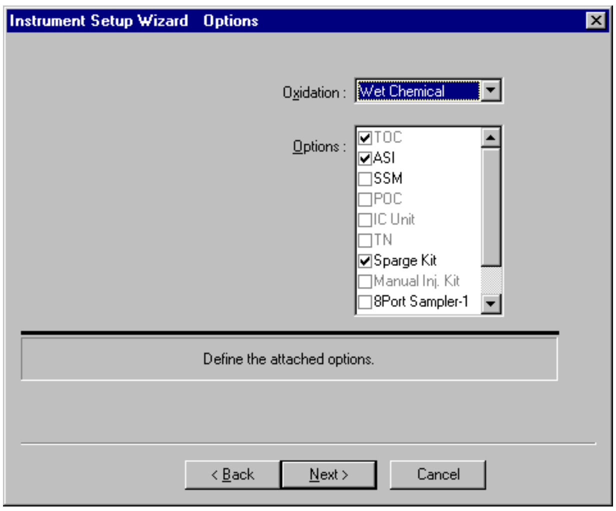


Figure 4.29 Instrument Setup Wizard: Options Window

7. Specify the wet chemical options that are configured on the system by clicking in the appropriate check boxes, as shown in the above figure.

8. Click the Next button. The TOC window of the Instrument Setup Wizard is displayed.

9. Select the TOC analysis parameters, as shown in the figure below.

Note: Be sure that the “Enable ready status check” option is selected. Enabling the ready status check function means that the instrument will not begin a new injection until the instrument status is Ready.

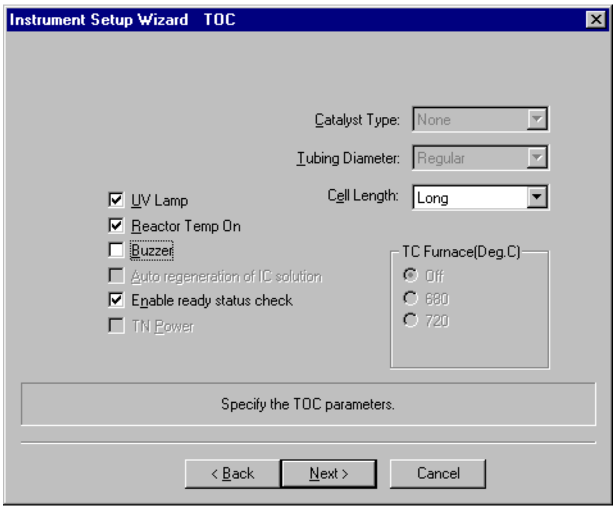


Figure 4.30 Instrument Setup Wizard: TOC Analysis Parameters

<u>Reference:</u> *Refer to Section 4.2.1 “TOC Parameter Settings” for details on TOC parameters.*

10. Click the Next button. The ASI window of the Instrument Setup Wizard is displayed. Verify that the ASI parameters match those shown on the figure below.

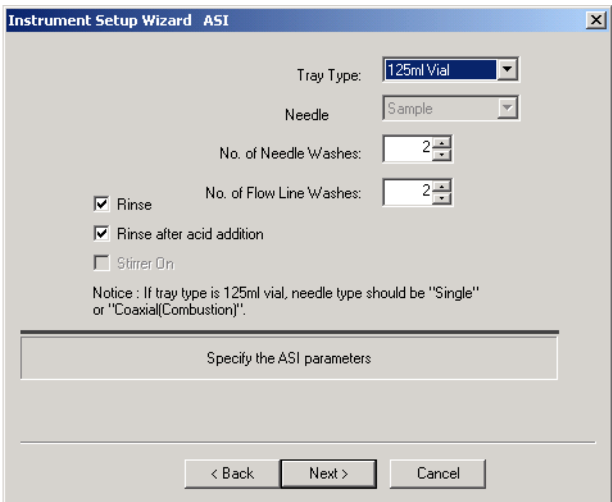


Figure 4.31 Instrument Setup Wizard: Autosampler Parameters

11. Click the Next button. The Communication window of the Instrument Setup Wizard is displayed.

12. Verify the communication information (COM Port, Stop Bits, and Parity).

    Note: If a Shimadzu representative installed your TOC-Control V software, the correct settings for your system are already configured. Do not change the settings. Refer to Section 3.2.6 "Communication Window" for more information on Communication settings.

13. Click the Next button. The History Function window of the Instrument Setup Wizard is displayed. If desired, enable the History Log to track all changes to the method. Refer to Section 3.2.7 "History Function Window" for more information.

    Note: The History Log function is automatically activated if Audit Trail was checked for the Security item in TOC-Control V Administrator window in the main menu.

14. Click the Finish button.

    An icon for the system now appears in the TOC-Control V main window.

TIP: *To edit the system configuration, double-click the system icon in the TOC-Control V main window or the Sample Table Editor window. Select Properties from the Instrument menu. The Instrument Properties dialog box opens, and system configuration options are displayed on tabs in the dialog box.*

(Topic 2) Connecting the Instrument

Opening a New Sample Table

1. In the TOC-Control V main window, double-click the Sample Table Editor icon. The User dialog box is displayed.

2. Enter your user name and password. Click the OK button.

3. Open a new Sample Table by either selecting New from the File menu or clicking the New toolbar button. The New dialog box is displayed.

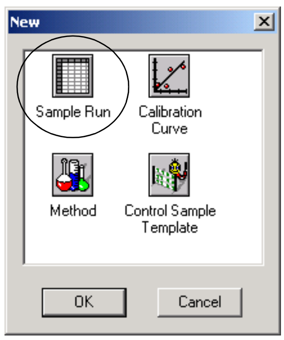


Figure 4.32 New Dialog Box

4. Select the Sample Run icon and click the OK button.

   The General Information dialog box opens.

5. Click the System tab and select the desired system from the drop-down list.

6. Save the Sample Table (.t32) file.

   Select Save File As in the File menu. Enter Tutorial and click the Save button.

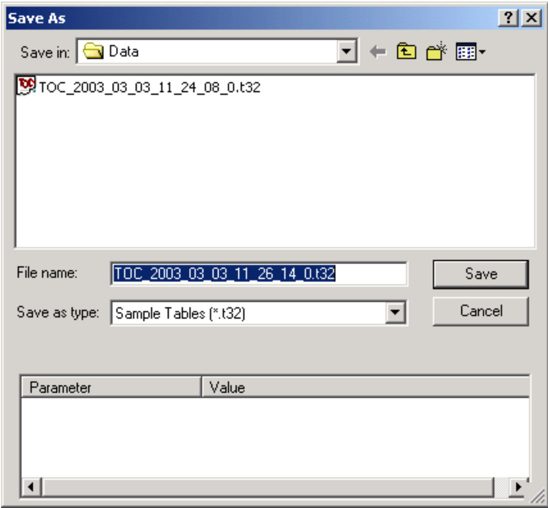


Figure 4.33 Save As Dialog Box

7. Click the OK button.

   A new, blank Sample Table is displayed.

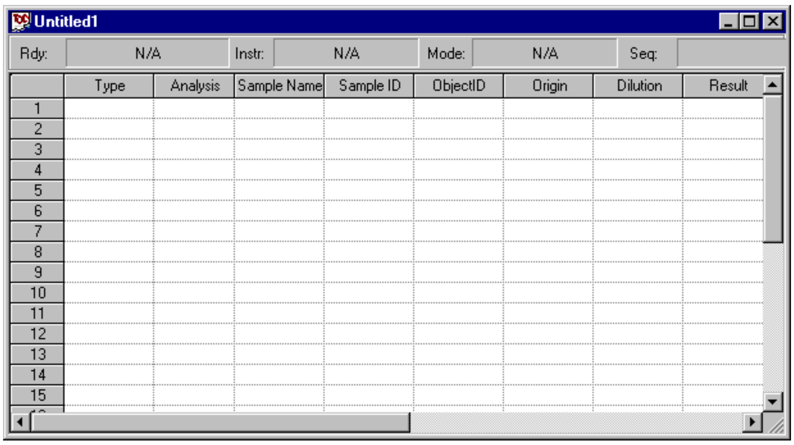


Figure 4.34 Sample Table

Connecting to the Instrument

1. To establish communication between the software and the instrument, either select Connect from the Instrument menu or click the Connect toolbar button.

The Parameter Configuration dialog box is displayed. Click the Use Settings on PC button.

The Sequence dialog box is displayed.

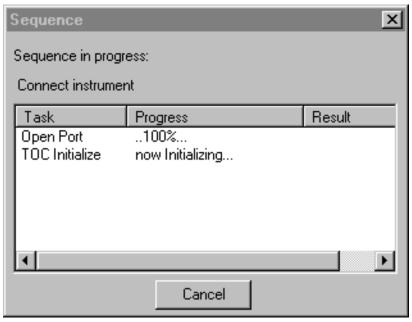


Figure 4.35 Sequence Dialog box

2. The Sequence dialog box displays information about the attempt to establish communication between the software and the instrument.

(Topic 3) Creating a Sample Table

Procedure

1. Enter the default analysis parameters for the Sample Table (.t32) file.

2. From the Options menu, select Default Measurement Parameters.

3. Three injections of each calibration standard and unknown sample are made. On the TC tab, change the “No. of injections” value to 3.

The other default measurement parameters should not be changed.

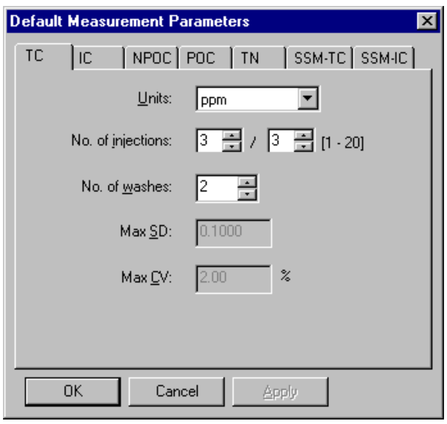


Figure 4.36 Default Measurement Parameters Dialog Box

Note: The selected measurement parameters will appear as default values when setting up calibration and method files.

Reference: *Refer to Section 4.2.3 "Setting Default Measurement Parameters" for details.*

4. Modify the Sample Table tab at the top of the window so that all items contained in the Sample Table are shown on the screen. The default display settings may not include all of the Sample Table parameters.

5. From the Options menu, select Display Settings>Table Options.

   Use the Select All button to ensure that all items on both tabs in the Table Options window are selected.

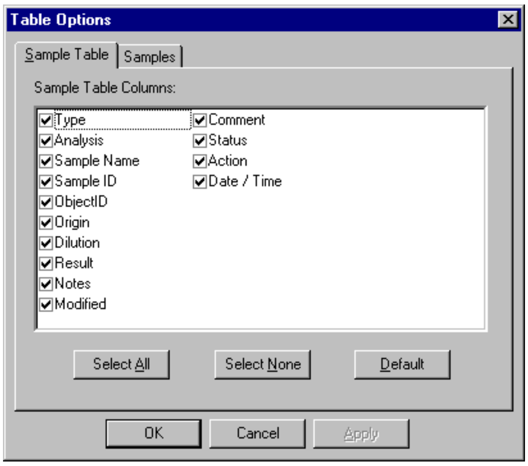


Figure 4.37 Table Options Window

Note: The selected measurement parameters will appear as default values when setting up calibration and method files.

Reference: *Refer to Section 4.4 "Sample Table Editor" for detailed descriptions of the Sample Table Editor menus and commands.*

(Topic 4) Setting up the Calibration Standard Runs

There are two steps to setting up calibration standard runs: creating a calibration file, which stores information about calibration standard measurements, and inserting the calibration standard runs in the Sample Table. Separate calibration files are created for the TC analysis. The calibration is a three-point curve generated using potassium hydrogen phthalate standard solutions.

Creating the TC Calibration File

1. Open the Sample Table Editor. From the File menu, select New.

   The New dialog box is displayed. Click the Calibration Curve icon.

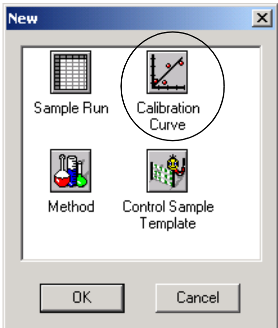


Figure 4.38 New Dialog Box

2. Click the OK button to open the Calibration Curve Wizard. Specific information about the calibration is entered on this page.

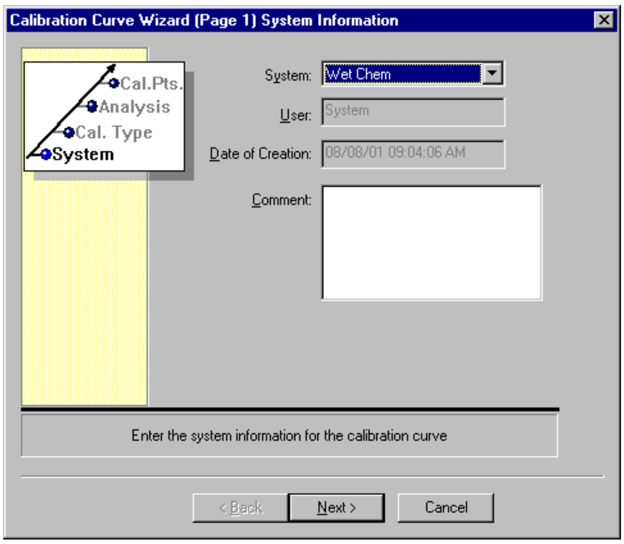


Figure 4.39 Calibration Curve Wizard (Page 1) System Information

3. Select the system to be used for the analyses from the drop-down list on page 1 System Information.

4. Click the Next button.

Page 2 of the Calibration Curve Wizard is displayed. Choose the type of calibration curve.

- Calibration points are distributed uniformly over the concentration range
- Edit calibration points manually
- Calibration curve according to DIN 38402/P-51
- Calibration curve according to USP/EP

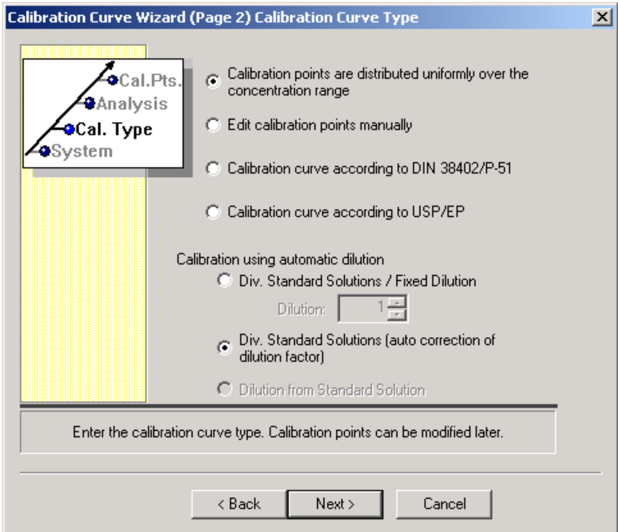


Figure 4.40 Calibration Curve Wizard (Page 2) Calibration Curve Type

5. Click the radio button for “Calibration points are distributed uniformly over the concentration range.” In the future, other options may be more appropriate. Refer to Section 4.4.2.2 “Calibration Curve” for a detailed description of the calibration curve types.

6. Click the Next button.

Page 3 of the Calibration Curve Wizard is displayed. The following options for calibration analysis are displayed:

Set the following calibration curve analysis parameters:

- Analysis: TC
- Default Sample Name: Standard
- Default Sample ID: Tutorial
- Calculation Method: Linear Regression
- Check box for Zero Shift: Yes (On)
- Calibration File Name: *TCTutorial.cal*

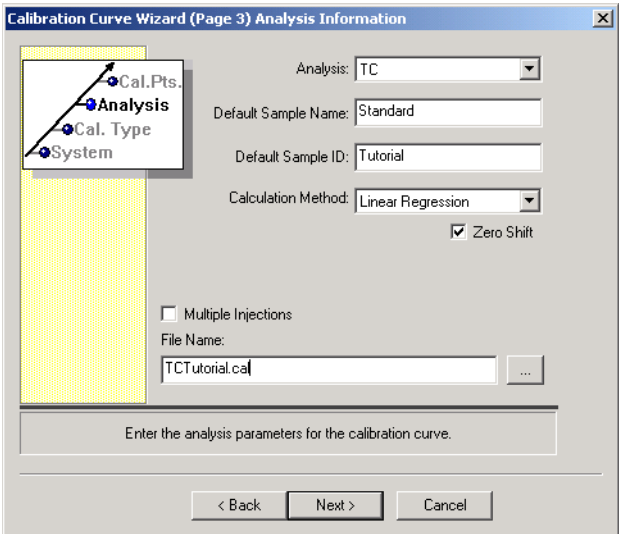


Figure 4.41 Calibration Curve Wizard (Page 3) Analysis Information

7. Click the Next button.

Page 4 of the Calibration Curve Wizard is displayed.

The No. of Injections matches the number entered in the default measurement parameters for the Sample Table. Refer to Step 3 of this tutorial for more information.

8. Enter the TC calibration standard concentration range values of 0 to 10mg/L.

Enter 3 as the number of calibration points.

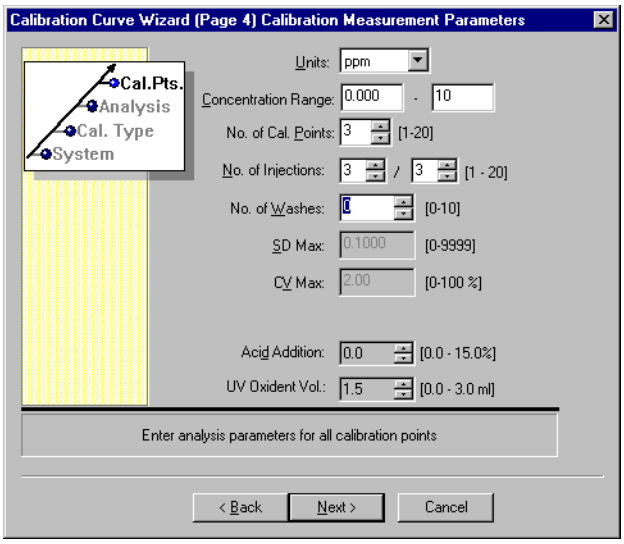


Figure 4.42 Calibration Curve Wizard (Page 4) Calibration Measurement Parameters

9. Click the Next button.

Page 5 of the Calibration Curve Wizard is displayed.

The Calibration Points List was completed based on information entered on the previous Wizard pages.

10. To make changes, select a row and click the Edit button.

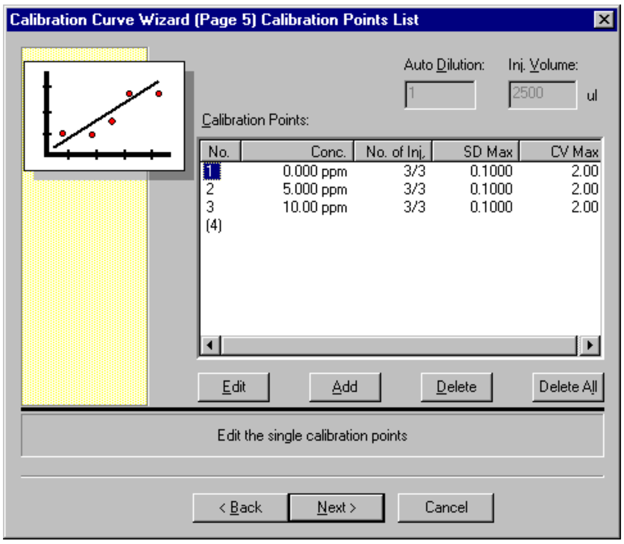


Figure 4.43 Calibration Curve Wizard (Page 5) Calibration Points List

11. Click the Next button.

Page 6 of the Calibration Curve Wizard is displayed.

12. Settings can be made on this page to delay or extend the peak detection time. View but do not change the default setting on the page.

13. Click the Next button.

Page 7 of the Calibration Curve Wizard is displayed. The History function may be enabled on this page. View but do not change the default setting.

14. Click the Finish button.

The Calibration Curve Wizard closes. The TC calibration curve file is now complete and has been saved.

Entering the Calibration Standard Runs in the Sample Table

Now that the calibration file has been created and saved, the calibration standard runs must be inserted into the Sample Table.

1. Insert the TC calibration standard runs by placing the cursor in the first line of the Sample Table.

2. From the Insert menu, select Calibration Curve.

3. Specify the name of the TC Calibration Curve (*tctutorial.cal*) and click the Open button.

The Sparging/Acid Addition window is displayed. Notice that the TC calibration standards have been automatically added to the table.

4. Enter the vial positions (1-3) for the calibration standards directly into the Vial column of the table. As vial positions are entered in the table, they are highlighted on the tray diagram in the Sparging/Acid Addition window.

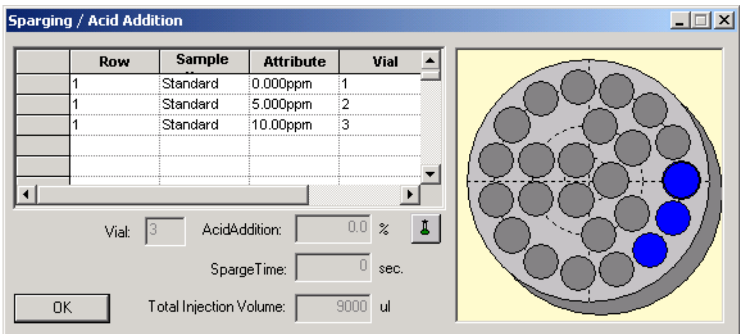


Figure 4.44 Sparging/Acid Addition Window for the ASI-V Autosampler

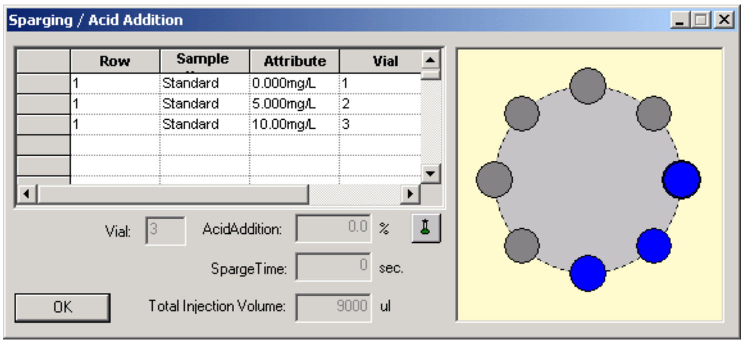


Figure 4.45 Sparging/Acid Addition Window for the 8-Port Sampler

5. Verify the information, then click the OK button. The Sample Table is displayed.The first row of the Sample Table contains the complete TC calibration standard measurement information.

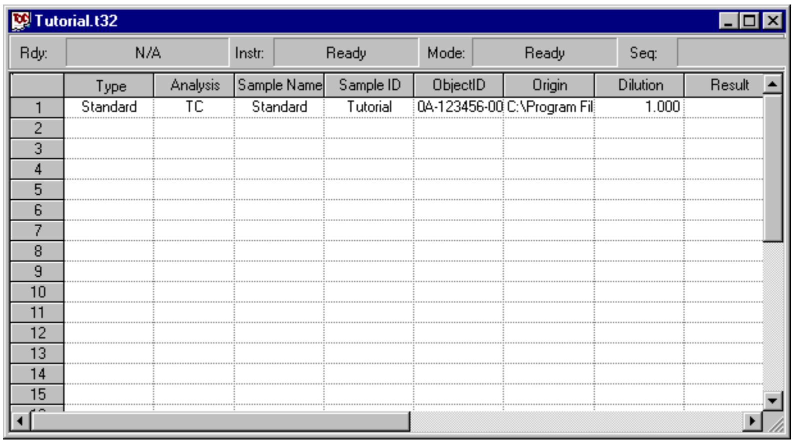


Figure 4.46 TC Calibration Standard Information in the Sample Table

6. Save the Sample Table by selecting Save from the File menu or by clicking the Save button in the toolbar.

(Topic 5) Setting Up an Unknown Sample Run

In Topic 4 of the tutorial, you learned that calibration standard measurement information is saved in a calibration file. You also learned that setting up a calibration standard run involves two steps: creating a calibration file and then inserting the calibration standards into the Sample Table.

By contrast, information about unknown sample measurement is saved in a method file. Setting up an unknown sample run involves two steps: creating a method file and then inserting the unknown sample into the Sample Table. In this tutorial, one “unknown” sample is analyzed. The “unknown” sample is actually the 5mg/L phthalate solution.

Creating a Method File

1. Open the Sample Table Editor. From the File menu, select New.

2. The New dialog box is displayed. Select the Method icon and click the OK button. The Method Wizard opens.

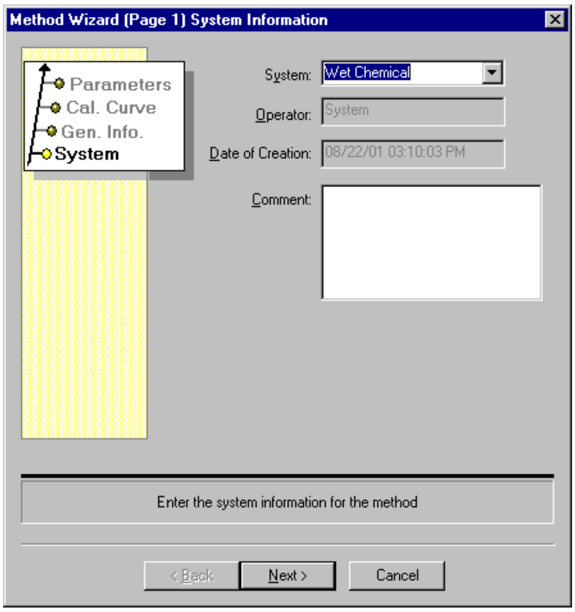


Figure 4.47 Method Wizard (Page1) System Information

3. Select the system to be used for the analysis from the drop-down box. Click the Next button.

   Page 2 of the Method Wizard is displayed.

4. Select TC from the Analysis drop-down menu. Enter “Sample” in the Default Sample Name field and “Tutorial” in the Default Sample ID field. In the File Name field, enter “*Tutorial.met*” as the name of the Method File.

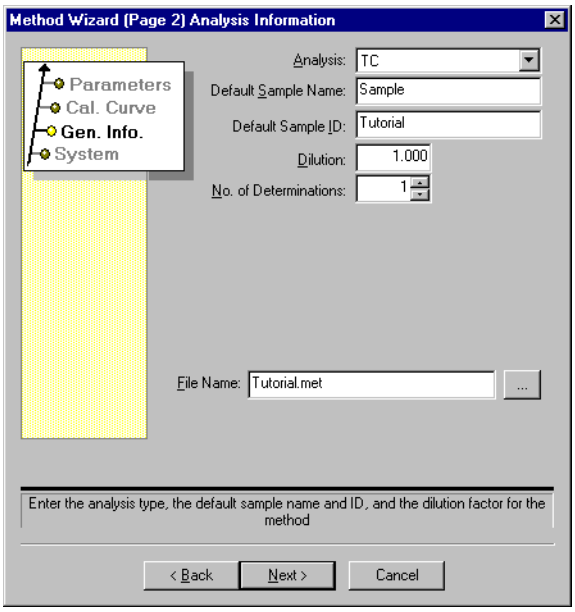


Figure 4.48 Method Wizard (Page 2) Analysis Information

5. Click the Next button.

Page 3 of the Method Wizard is displayed.

Note: It is possible to enter the calibration curve file in this screen since it contains the relevant data.

6. Place the cursor in the Calibration Curve 1 field. Click the Browse ... button.

Select the file for the TC calibration curve created earlier (*TCtutorial.cal*).

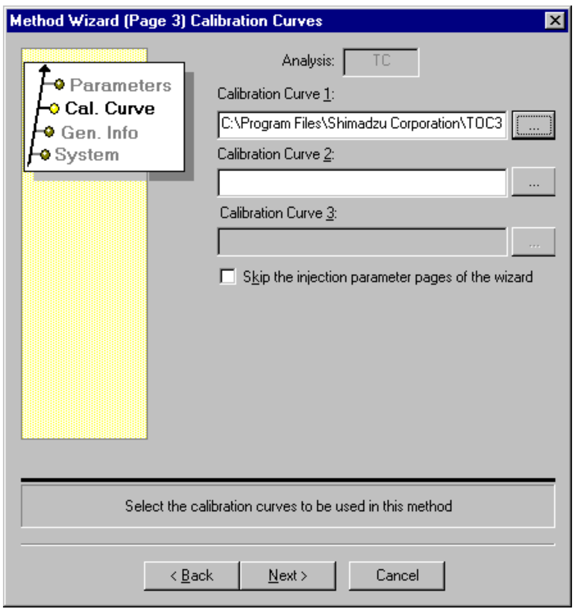


Figure 4.49 Method Wizard (Page 3) Calibration Curves

7. Click the Next button.

Page 4 of the Method Wizard is displayed.

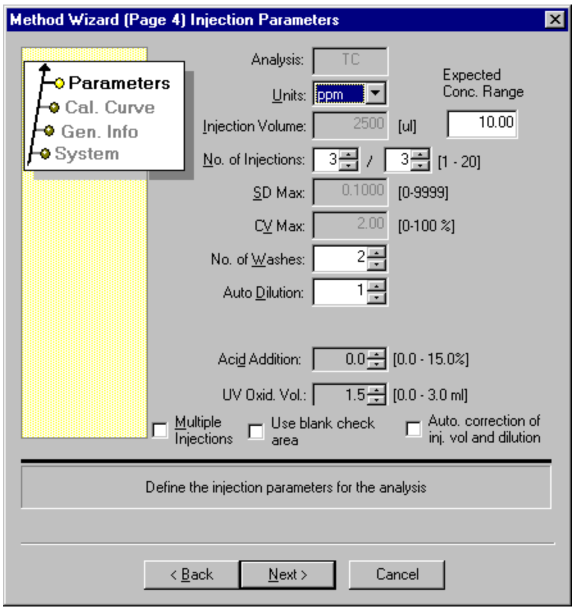


Figure 4.50 Method Wizard (Page 4) Injection Parameters

8. The following TC analysis parameters are entered.

- Analysis (disabled)
- Units
- Injection Volume (disabled)
- Expected Conc. Range
- No. of Injections
- SD Max
- CV Max
- No. of Washes
- Auto Dilution
- Acid Addition
- UV Oxid.Vol
- Use blank check area
- Auto correction of inj. vol and dilution

9. Click the Next button.

Page 5 of the Method Wizard is displayed. View but do not change the default settings on this page.

10. Click the Next button.

    Page 6 of the Method Wizard is displayed. View but do not change the default settings on this page.

11. Click the Next button.

    Page 7 of the Method Wizard is displayed. View but do not change the default settings on this page.

12. Click the Finish button.

    The Method Wizard closes. The method file is now complete and has been saved. Insert the sample into the Sample Table.

Reference: *Refer to Section 4.4.2.3 "Method Wizard" for a detailed description of the Method Wizard.*

13. Insert the unknown sample by placing the cursor in the second line of the Sample Table. From the Insert menu, select Sample.

    Page 1 of the Sample Wizard (Parameter Source) is displayed.

TIP: *To set up multiple analyses using a previously created method, select the Auto Generate Table command from the Insert menu. Refer to Section 4.4.5.1 "Auto Generate" for details.*

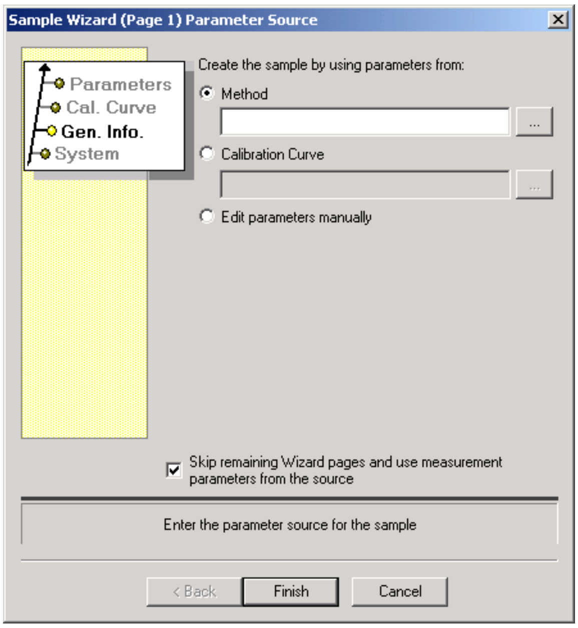


Figure 4.51 Sample Wizard (Page 1) Parameter Source

14. Click the Method radio button. By selecting this option, the sample is analyzed using the measurement parameters specified in the method just created. Enter the name of the method file (*Tutorial.met*) or click the Browse button to search for the file.

    Click in the "Skip remaining Wizard pages and use measurement parameters from the source" check box. By selecting this option, the analysis parameters for the unknown sample will automatically be copied from the source (method) file. As a result, the parameters do not need to be entered manually and the remaining pages of the Sample Wizard are not displayed.

15. Click the Finish button.

    The Sparging/Acid Addition window is displayed.

16. Enter the vial position (2) for the unknown sample (5mgC/L standard) directly into the Vial column of the table.

17. Verify the information and click the OK button.

    The second row of the Sample Table now contains the measurement parameters for the unknown sample. (A value is not displayed in the Result column until measurement is complete.)

18. Save the Sample Table by selecting Save from the File menu or by clicking the Save button in the toolbar.

Measurement can now be performed.

(Topic 6) Performing Measurement

Procedure

1. Prepare the three calibration standards (0mg/L, 5mg/L and 10mg/L potassium hydrogen phthalate) and place them in autosampler tray vial positions 1, 2, and 3.

   Note: Calibration standards and unknown samples can be placed in any vial position when controlling the instrument with the TOC-Control V software.

2. Verify the status of the instrument detectors before starting analysis. From the Instrument menu, select Background Monitor, or click the Background Monitor button on the toolbar.

   The Background Monitor window is displayed. This window is used to monitor the real time signal from the ASI and the detectors. On the TOC tab, the status of the baseline should be OK for position, fluctuation, and noise.

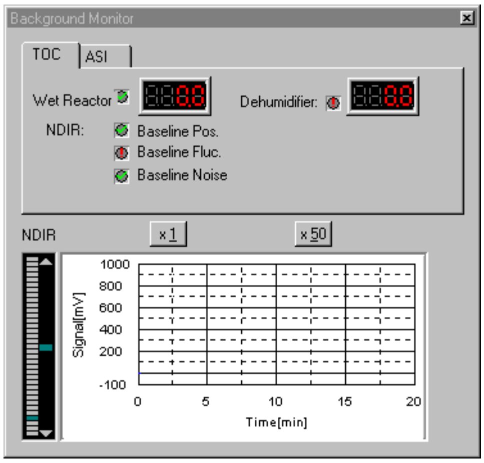


Figure 4.52 Background Monitor Window: TOC Tab

3. Since analysis cannot be performed with the Background Monitor open, close the window.

4. Place the cursor in the first row of the Sample Table. From the Instrument menu, select Start, or click the Start button on the toolbar.

   The Standby window is displayed.

5. Select Keep running from the options on the Standby window.
   - Keep running
   - Shut down instrument
   - Auto restart

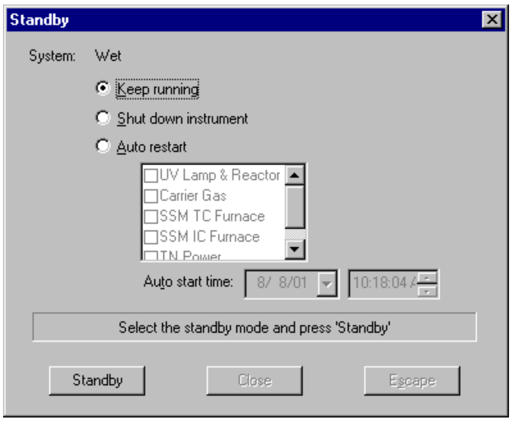


Figure 4.53 Standby Window

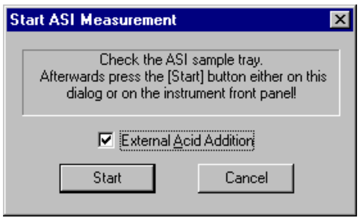
Reference: *Refer to Section 4.4.6.3 "Standby" for detailed descriptions of the Standby window options.*

6. Click the Standby button.

   The Sparging/Acid Addition window is displayed.

7. Verify the vial positions and click the OK button.

   The Start ASI Measurement window is displayed.



8. Click the Start button to begin the analysis.

9. The status of the measurements can be monitored during analysis by viewing the Sample Window. To open the Sample Window, either select Sample Window from the View menu or select the Sample Window button on the toolbar.

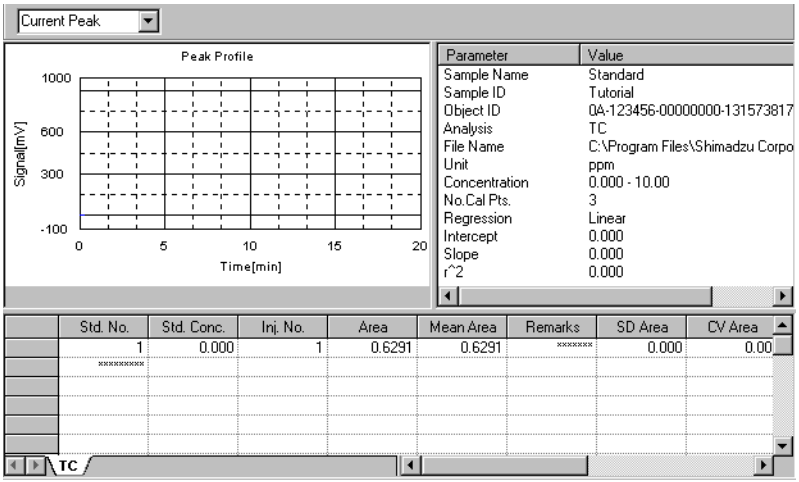


Figure 4.54 Sample Window

Note: Sample Window

- The Sample Window is divided into 3 sections, as shown in the above figure. The Graph is in the upper left corner, the Parameter Table is in the upper right corner, and the Injection Table is the bottom half of the window. The current injection is displayed with asterisks in the Injection Table. As each measurement occurs, information in the appropriate Injection Table row is completed. Previous injections are shown above the current row. Use the scroll bar to see all of the information.
- To view the peak profile of the measurement in progress, click the asterisk of Sample No. To view a previous measurement, click that row.
- When the Sample Window is opened, the text on the graph may be overly enlarged. In that situation, close the Sample Window and then open it again.
- During measurement, even if “All Peaks” is selected, because it takes time for the screen to update, the peak display may not change. In this case, select “All Peaks” following measurement.

10. Statistical information about the current set of injections is calculated and displayed in the Sample Window. Preliminary decisions about the measurements can be made while the analysis is still in progress.

Note: Information about the current injection is shown in the status box in the upper right portion of the Sample Window.

TIP: *The drop-down list above the graph is used to set the graph display. When Current Peak is selected, the peak profile of the current injection is drawn in the graph as the measurement is performed. Use this window to monitor the analysis as it occurs. To see the peak profile of a previous injection, click its row in the Injection Table. To see the peak profile of all the injections for a sample, select All Peaks from the drop-down list at the top of the graph. Use the right-mouse menu to customize the peak profile graph.*

Reference: *Refer to Section 4.4.4.3 "Sample Window" for a detailed description of Sample Window options.*

11. When measurement is complete, the autosampler moves to the rinse well for a flow line rinse and the instrument status tab displays Ready. Close the Sample Window.

(Topic 7) Evaluating the Results

The Sample Table now contains measurement information. Use the scroll bars to view the measurement information.

We will check the calibration curve results and evaluate the results for the unknown sample.

Checking the TC Calibration Curve Results

1. Place the cursor in the first row of the Sample Table.

2. From the View menu, select Calibration Curve, or click the Calibration Curve button on the toolbar.

   The Calibration Curve Properties window is displayed.

3. Select the Graph tab to view the TC calibration curve data in graph form.

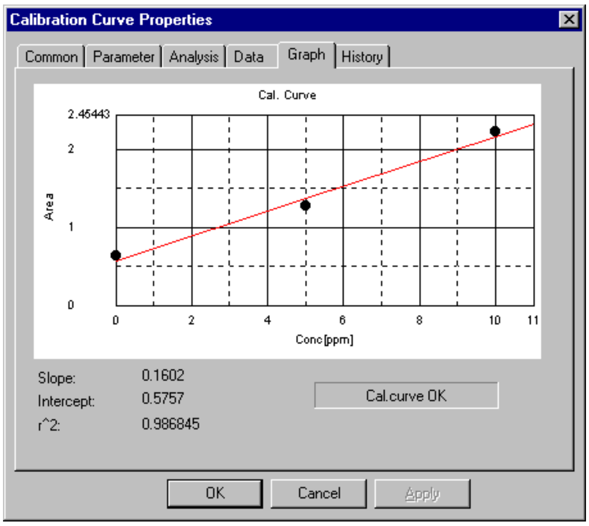


Figure 4.55 Calibration Curve Properties: Graph Tab

The coefficient of determination (r^2) value is a measure of the linearity of the relationship between concentration and area. Because we selected the linear regression calculation when we created the calibration file, an r^2 value was calculated for the TC calibration and displayed in the window. Use the r^2 value to determine the success of the calibration. An r^2 value of 1 indicates a perfectly linear relationship between concentration and area. Application methods may specify an acceptable limit for the correlation coefficient.

4. Select the Data tab to view calibration statistics such as Standard Deviation (SD) and Coefficient of Variation (CV). These statistics can also be used to evaluate the success of the calibration.

TIP: *A successful calibration can be used for future analyses by specifying the calibration file name (*.cal) in the method.*

5. Close the TC Calibration Curve Properties window when the evaluation is complete.

TIP: *When All Peaks is selected from the drop-down list at the top of the graph, clicking in another row of the Sample Table will automatically update the Sample Window display with the peak profiles for that row.*

Evaluating the Unknown Sample Results

1. Place the cursor in the second row of the Sample Table, which contains analysis information for the unknown sample.

2. Examine the concentration results by scrolling to the Result column in the Sample Window.

   Note: The results should be close to 5mg/L.

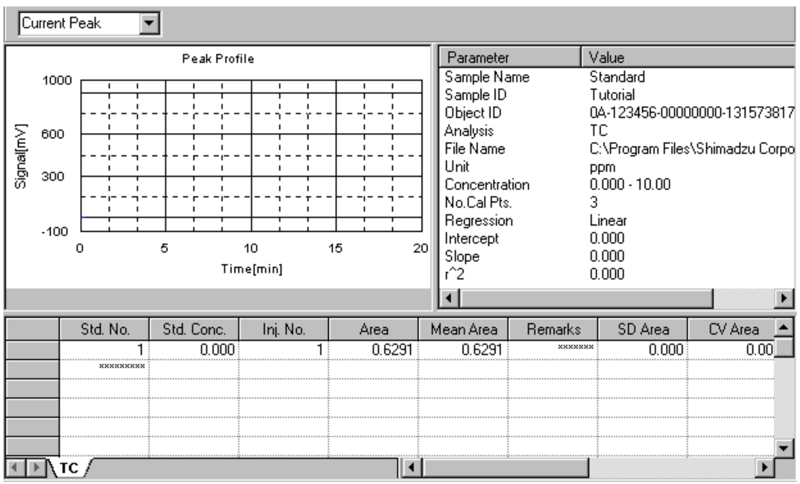


Figure 4.56 Unknown Sample Results

3. Close the Sample Window when the evaluation is complete.

Outlier Test

The Outlier Test is used to provide additional information about the measurement result.

1. Place the cursor in the second row of the Sample Table, which contains analysis information for the unknown sample.

2. From the View menu, select Outlier Test.

Note: The Outlier Test is conducted to identify suspect results. The test statistically compares all unknown samples that have the same sample name. Because this tutorial includes only one measurement of the unknown sample, the outlier test calculation is not meaningful.

(Topic 8) Printing a Report

Print a report after measurement is complete and the results have been evaluated.

Selecting the Report Items

1. From the File menu, select Page Setup.

2. The Page Setup window is displayed. Each tab in the window represents a section of the report and contains several items. Selections are made on each tab to print a typical report for the calibration standards and unknown sample.

Reference: *Refer to Section 4.4.2.15 "Page Setup" for detailed descriptions of the Page Setup options.*

3. On the Instrument tab, ensure that the *Print system information* check box is selected, then select the options shown in the figure below.

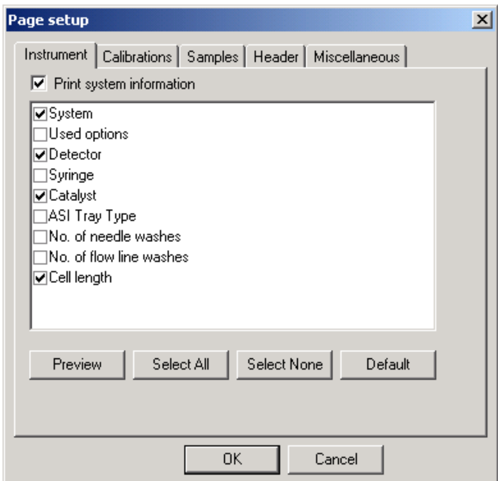


Figure 4.57 Page Setup Instrument Tab

4. Open the Calibrations tab and select the options shown in the figure below.

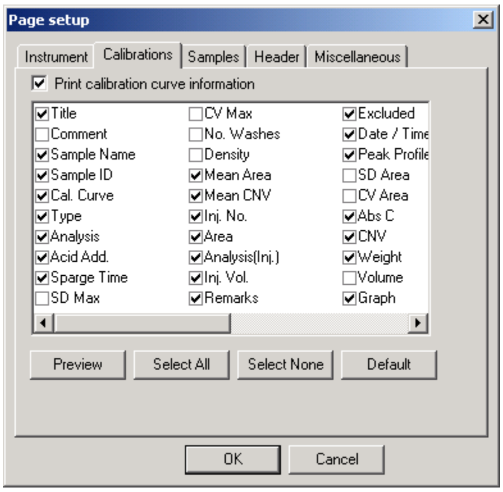


Figure 4.58 Page Setup: Calibrations Tab

5. Open the Samples tab and select the options shown in the figure below.

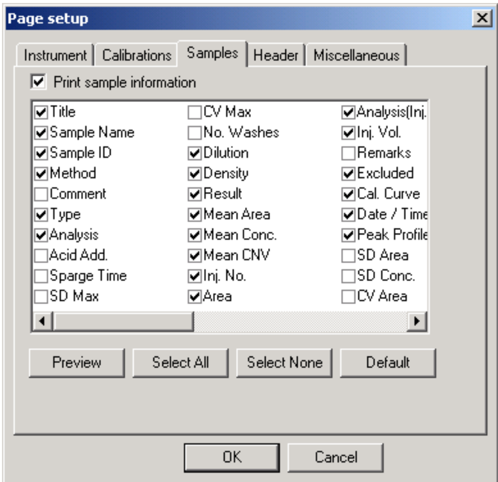


Figure 4.59 Page Setup: Samples Tab

Note: Use the default selections on the Header and Grid tabs.

6. Open the Miscellaneous tab and enter “Tutorial” in the Organization name field. Select the Footer check box to include page numbers on the report.

TIP: *A company logo can be added to the report header by selecting the Logo check box and specifying the logo graphic file.*

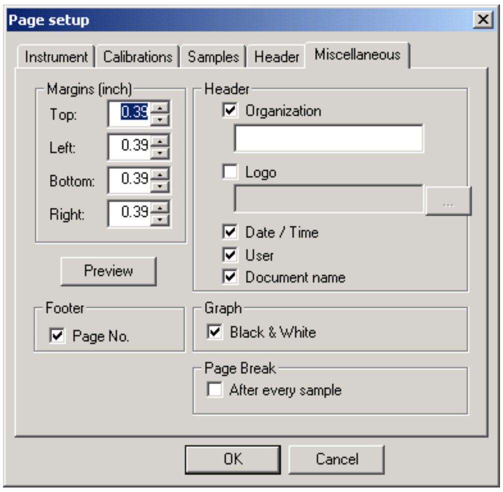


Figure 4.60 Page Setup: Miscellaneous Tab

Previewing the Report

1. Select the Preview button from any of the Page Setup tabs to preview the entire report before printing.

TIP: *The report can also be previewed from outside the Page Setup window by selecting Print Preview>Sample Report from the File menu.*

The Preview feature displays each page of the report exactly as it is printed. Notice that each item that was selected in the Page Setup window appears in the report.

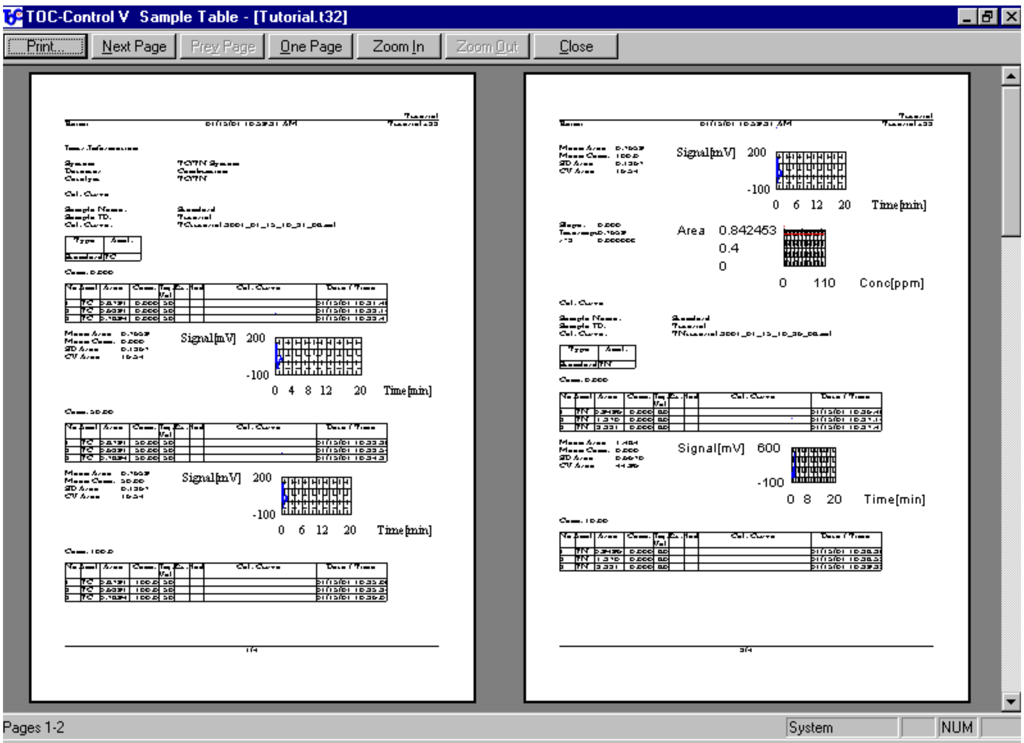


Figure 4.61 Preview the Report Appearance

TIP: *Magnify the view by clicking the magnifying glass tool, or on the Zoom In button. Move from page to page by clicking the Next Page and Previous Page buttons.*

2. Print the report by clicking the Print button.

This ends the tutorial.

Reference: *The tutorial provided instructions for setting instrument configuration and analysis parameters, adding calibration standard and unknown sample information to the Sample Table, creating calibration curves and method files, conducting measurements, and checking and printing results. More details on all of these procedures are discussed in* *Section 3.2 "Instrument Setup and System Properties", Section 4.2 "Setting General Measurement Parameters", and Section 4.4 "Sample Table Editor".*

4.3.3 TC Blank Check Analysis

A TOC system invariably contains substances in the instrument flow lines that produce peaks. These peaks are referred to as blank values or system blank values, and are present even in water samples containing absolutely no carbon. The magnitude of peaks in the blanks differs based on various factors. These peaks can affect the accuracy of the TC analyses. For the TOC-V, every effort has been made to minimize these peaks. Accuracy is particularly affected when measuring samples such as ultra-pure water which have trace amounts of TOC. The blank peaks must be small and stable. To check and correct for the magnitude of the system blank peaks, use the TOC-Control V Blank Check procedure described below.

The magnitude of the blank and the instrument stability can be assessed from the area values obtained during blank measurement. Compare the area of the blank to the area determined from the calibration curve to determine the equivalent concentration value for the blank.

Note:

- The Blank Check procedure takes about 6 hours to complete and does not require continuous monitoring by the user. As a result, performing the procedure overnight is recommended.
- Blank values measured during the Blank Check procedure should rapidly decrease in magnitude and stabilize. If the instrument flow lines, syringe pump interior, or reactor have been contaminated, the blank values will not rapidly decrease. In such instances, the Blank Check procedure should be repeated.
- Blank peaks are negligible in the IC analysis flow line. The IC value is zero if the water is acidified and sparged. This can easily be confirmed by conducting analysis using the IC flow line; however, this is not normally necessary. Consequently, there are no special IC flow line check procedures, as with the TC blank check.

Blank Check Procedure

1. Open the TOC-Control V Sample Table Editor and create a blank Sample Table for the high sensitivity system.

   Note: Do not insert a sample into the Sample Table. The Blank Check procedure only works with an empty Sample Table.

2. Connect the instrument by selecting Connect from the Instrument menu.

3. From the Instrument menu, select Maintenance>Blank Check.

   The Blank Check Parameters dialog box is displayed.

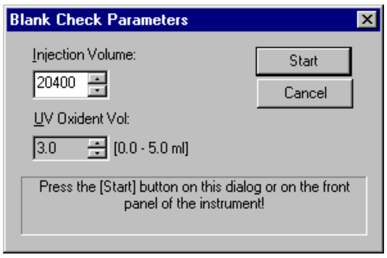


Figure 4.62 Blank Check Parameters Dialog Box

4. Select the injection volume using the spin controls in the Injection Volume field. Select the volume of oxidizer solution using the spin controls in the UV Oxident Vol. field. Click the Start button to display the Standby dialog box.

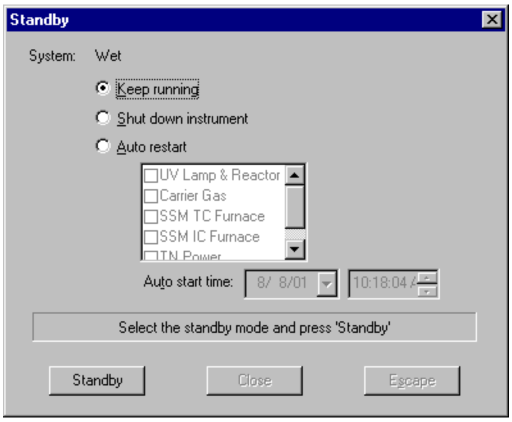


Figure 4.63 Standby Dialog Box

5. Select an option to determine the condition of the instrument after the blank check is complete. Options are:
   - Keep running
   - Shut down instrument
   - Auto restart (UV lamp, reactor temperature on, carrier gas, restart time)

6. Insert the sampling tube into a container with at least 300mL of purified water.

7. Click the Standby button.

   The Save As dialog box is displayed.

8. Enter a file name and click the Save button. Data from the Blank Check procedure is stored in the created file.

   Blank check analysis begins.

4.3.4 Ending Measurement

Follow the procedure below to end measurement.

Procedure

1. From the Instrument menu, select Standby.

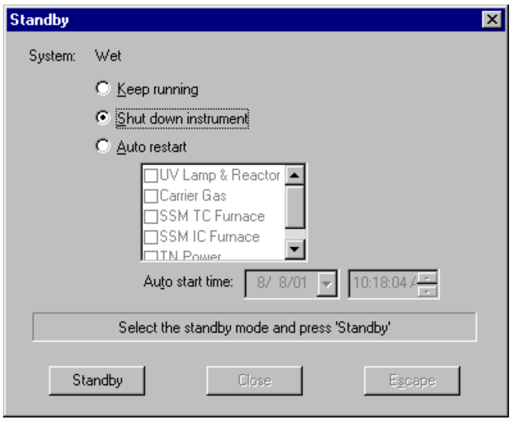


Figure 4.64 Standby Window

2. Two procedures are available for ending measurement:
   - Shut down instrument
   - Auto restart

   These procedures are described below.

Shut Down Instrument

Use the “Shut down instrument” option to end measurement and switch off power to the instrument.

1. Select “Shut down instrument” in the Standby window and click the Standby button.

Power is automatically cut to the TC reactor, UV lamp and heater, carrier gas flow is stopped, and power to the instrument is turned off.

Note: When “Shut down instrument” is selected, the instrument power is turned off and the power to the autosampler exhaust fan is turned off. If acidic samples are being measured or if sparging is being performed using the autosampler in NPOC measurement, there is a danger that following measurement, volatilization of acid in the sample could cause corrosion of the autosampler. Do not switch off the power to the instrument. (Do not select Shutdown.)

Auto Restart

The Auto Restart function is used to shut down the instrument and then restart it at a specified time. Selections are made for the date and time that measurement will next be performed, as well as for the instrument restart settings. After shutting down the instrument, the carrier gas flow and power to the TC reactor UV lamp and heater are automatically turned off. Approximately 30 minutes before the restart time, the UV lamp is turned on and the heater temperature begins to rise. Carrier gas is automatically turned on to allow the instrument to achieve a Ready state by the restart time.

1. Select “Auto restart” in the Standby window.

2. Select the appropriate instrument restart settings (UV lamp, Reactor temp On and Carrier gas). Set the restart date and time.

Note: If a check is placed in the box beside a unit, the power remains on and the carrier gas continues to flow. In order to have the power turned off automatically, do not place a check mark in any of these boxes.

3. Click the Standby button.

Power to the instrument is automatically turned off and will be automatically turned on about 30 minutes before the restart date and time. By using Auto Restart, the instrument is Ready for measurement at the specified startup time.

Reference: *Refer to Section 4.4.6.3 “Standby” for detailed descriptions of the available instrument shutdown options.*

4.4 Sample Table Editor

The TOC-Control V Sample Table Editor menus and commands are described in this section.

4.4.1 Sample Table Window Overview

4.4.1.1 Toolbar Functions

If the Toolbar is not visible, select Toolbar from the View menu, then specify the desired Toolbar options.

Note: Many of the Toolbar functions are disabled until a Sample Table is opened or communication is established with an instrument.

TIP: *A description of each Toolbar button's function appears when the mouse pointer is positioned over the button.*
All of the Toolbar commands are also accessible through the Sample Table Editor menus.

New File

Opens a new, blank Sample Table.

Open File

Opens a saved Sample Table. Click the button to display the Open dialog box. Select a *.t32 file.

Save File

Saves the data in the open Sample Table. If the open Sample Table is new and has not yet been saved, the Save As dialog box is displayed.

Cut

Removes data from highlighted cells in a table and saves them to the clipboard.

Copy

Copies data from highlighted cells in a table and saves them to the clipboard.

Paste

Copies the contents of the clipboard to a selected location in a table.

Print

Sends the data from the currently displayed window to the printer. Clicking the button starts printing immediately using the current printer settings.

Help - About

Provides information about the TOC-Control V software.

Background Monitor

Displays the Background Monitor window, which displays the status of the ASI, TC reactor and the instrument detector(s).

Connect

Opens or closes the electronic connection between the instrument and the PC.

Standby

Shuts down or reactivates the instrument.

Start/Continue

Starts measurement. Either the Manual Injection Parameter or Sparging/Acid Addition window is displayed, depending on the instrument settings.

Stop

Stops the measurement sequence after all injections of the current sample are complete.

Halt

Immediately stops the current measurement and discards data from the interrupted measurement.

Peak Stop

Stops the measurement sequence and aborts analysis of the current sample. The peak in progress is treated as if it were a finished analysis.

Properties

Displays the Sample/Method Properties dialog box.

Calibration Curve (View)

Displays the calibration curve data. Highlight the calibration standard row in a Sample Table to select the calibration curve to examine.

Sample

Opens the Sample Window, which displays detailed information for each sample injection and can be used to view measurement data in real time.

Method

Opens the Method Properties window, which contains the method parameters for a selected sample.

Exclude

Excludes a selected sample or injection from the calculation. Toggles to include an excluded sample.

Recalculate Highlighted

Recalculates the highlighted sample results after applying a new calibration curve or excluding injections.

Recalculate All

Recalculates all sample results after applying a new calibration curve or excluding injections.

Delete Highlighted Data

Deletes the highlighted measurement data. This function does not change the structure of the Sample Table.

Delete All Data

Deletes the measurement data for all samples. This function does not change the structure of the Sample Table.

Format

Displays the Floating Point Format dialog box, where options for numerical formats can be modified.

Font

Displays the Font dialog box, where options for fonts can be modified.

Insert Sample

Opens the Sample Wizard, and allows a sample to be inserted in the active position of the Sample Table.

Autogenerate

Opens the Sample Group Wizard, and allows a specified group of samples to be inserted in the active position of the Sample Table.

Insert Control

Displays the Open dialog box, and allows a selected control sample template to be added to the Sample Table.

Insert Calibration Curve

Displays the Open dialog box, and allows a selected calibration curve to be added to the Sample Table.

4.4.1.2 Status Bar and Notification Bar

The Status Bar displays the status of various software functions and is displayed at the bottom of the open window for all applications. If the Status Bar is not visible, select Status Bar from the View menu.

The Notification bar displays information about the status of the TOC-Control V software, the connection between the instrument and the PC, measurement events, and operation errors. Use the Options>Display Settings>Notification bar Settings window to enable, disable, or select settings for the Notification bar. Refer to Section 4.4.9.5 "Display Settings>Notification Bar Settings" for details.

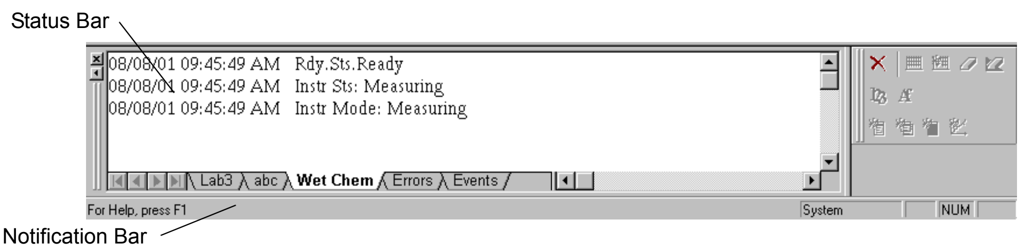


Figure 4.65 Status Bar and Notification Bar

4.4.2 File Menu

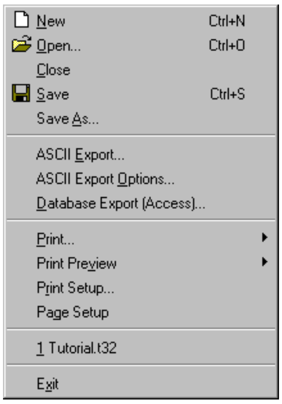


Figure 4.66 File Menu

4.4.2.1 New

The New command opens the New dialog box, which displays the options for all new file types. The options are Sample Run, Calibration Curve, Method and Control Sample Template.

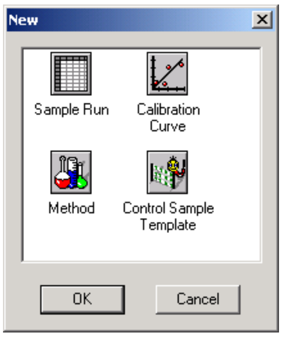


Figure 4.67 New Dialog Box

TIP: *A new file can also be created by clicking the New File button on the Toolbar and typing in the new file name.*

Sample Run

Displays the General Information window, which can also be accessed by selecting General Information from the Options menu. Refer to Section 4.4.9.1 "General Information" for a detailed description of the General Information window.

Calibration Curve

Opens the Calibration Curve Wizard, which can also be accessed by selecting Calibration Curve from the Insert menu. Refer to Section 4.4.2.2 "Calibration Curve" for a detailed description of the Calibration Curve Wizard.

Method

Opens the Method Wizard which can also be accessed by selecting File>New>Method. Refer to Section 4.4.2.3 “Method Wizard” for a detailed description of the Method Wizard.

Control Sample Template

Opens the Control Sample Wizard which can also be accessed by selecting File>New>Control Sample Template. Refer to Section 4.4.5.3 “Control” for a detailed description of the Control Sample Wizard. Refer to Appendix B for a description of the Control Charts function.

OK Button

Opens the file associated with the selected icon, and closes the New dialog box.

Cancel Button

Closes the New dialog box without making any changes.

4.4.2.2 Calibration Curve

The Calibration Curve command is used to insert a calibration curve into the Sample Table. The File>Open dialog box is displayed when the Calibration Curve command is selected.

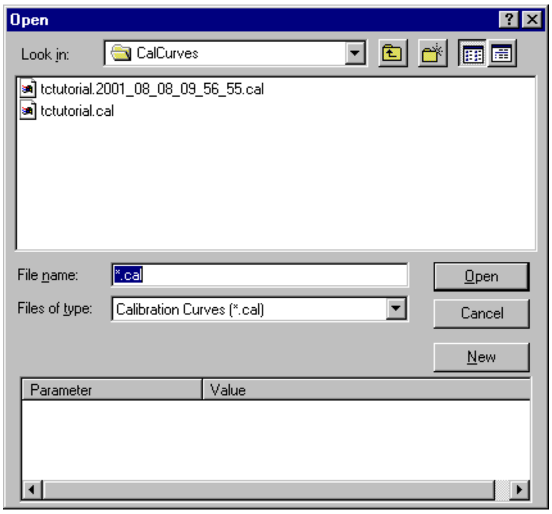


Figure 4.68 File>Open Dialog Box

A table of calibration information is displayed in the window at the bottom of the Open dialog box after a calibration curve is selected. Click the Open button to insert the selected calibration curve into the Sample Table. Click the Cancel button to abort the operation.

Note: The Insert>Calibration Curve command is not available during sample analysis.

- An error message is displayed if the selected file does not have the correct format or is not a calibration curve.

To create a new calibration curve for insertion into the Sample Table, click the New button to open the Calibration Curve Wizard. The Calibration Curve Wizard consists of several windows, each of which defines specific parameters for the calibration curve, as described below.

TIP: *The Calibration Curve Wizard can also be accessed by selecting File>New and double-clicking the Calibration Curve icon in the New dialog box.*

Calibration Curve Wizard (Page 1) System Information

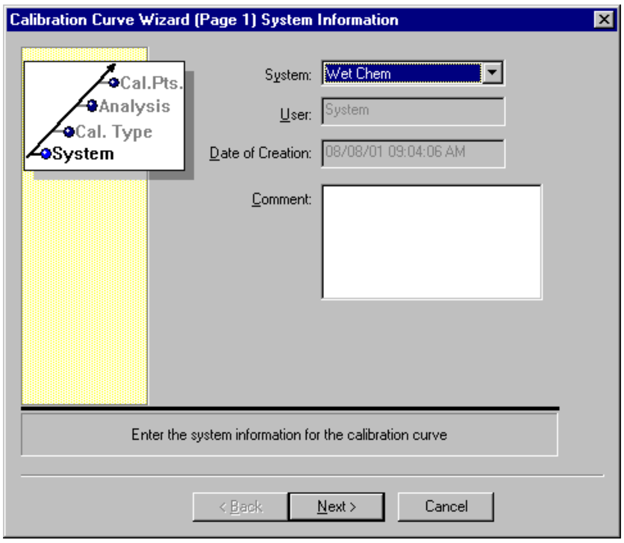


Figure 4.69 Calibration Curve Wizard (Page 1) System Information

Parameter	Description
System	Select the system to be used.
User	Displays the user name. This field cannot be edited.
Date of Creation	Displays the current system date and time. This field cannot be edited.
Comment	Enter a comment (512 characters maximum).

Calibration Curve Wizard (Page 2) Calibration Curve Type

The Calibration Curve Type window is used to specify the calibration curve type.

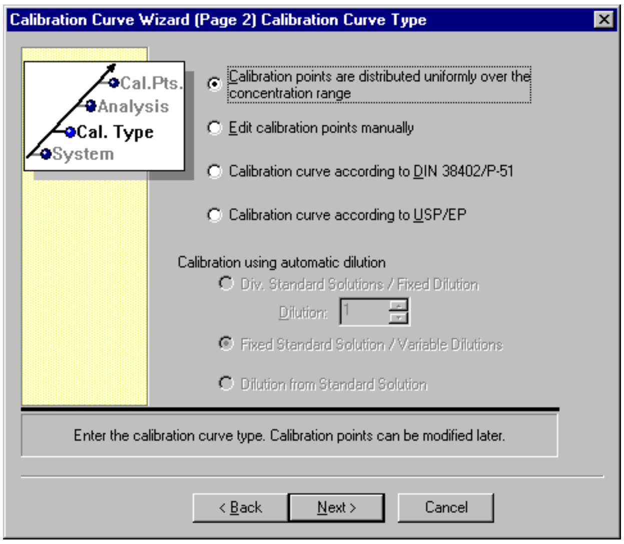


Figure 4.70 Calibration Curve Wizard (Page 2) Calibration Curve Type

Parameter	Description
Calibration points are distributed uniformly over the concentration range	Select this option to calculate the calibration point concentrations automatically. The software will distribute the points evenly over the calibration curve range and display these values on the next page.
Edit calibration points manually	Select this option to enter the calibration point concentrations manually on the Calibration Points List Wizard page.
Calibration curve according to DIN 38402/P-51	Select this option to create a calibration curve according to DIN 38402.
Calibration curve according to USP/EP	Select this option to create a calibration curve that consists of 2 calibration standards with concentrations between 0 and 500µg/L and one control sample with a concentration of 500µg/L.

Calibration using automatic dilution

Dilution Factor Manual Setting	For manually setting dilution factor of standard solution. Enter the dilution factor.
Dilution Factor Automatic Setting	Automatically calculates dilution factor for standard solution, and performs measurement using that dilution factor.
Use Source Dilution Function	Prepares standard solution automatically inside the instrument, and performs measurement using that diluted standard solution.

Calibration Curve Wizard (Page 3) Analysis Information

The Analysis Information window is used to specify the type of analysis and the standard name for the calibration curve.

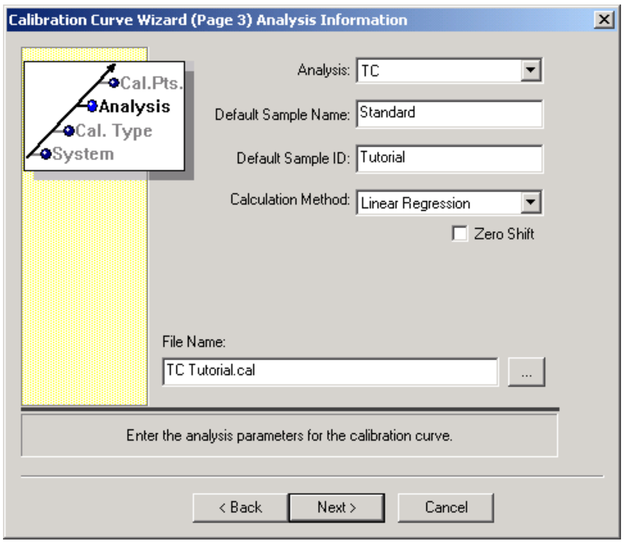


Figure 4.71 Calibration Curve Wizard (Page 3) Analysis Information

Parameter	Description
Analysis	Select the analysis type.
Default Sample Name	Enter the sample name for the calibration standards (64 characters maximum).
Default Sample ID	Enter the default sample identification for the calibration standards (64 characters maximum).
Calculation Method	Select the calculation method to be used for the calibration from the drop-down list. Available calculation methods are point-to-point and linear regression. Refer to Section 4.4.4.5 "Properties" for detailed descriptions of the calibration methods.
Zero Shift	Select this option to shift the calibration curve through the origin. Refer to Section 4.4.4.5 "Properties" for a detailed description of the Zero Shift option.
File Name	Enter the file name of the calibration curve.

Calibration Curve Wizard (Page 4) Calibration Measurement Parameters

The Calibration Measurement Parameters window is used to specify measurement parameters for the calibration standards.

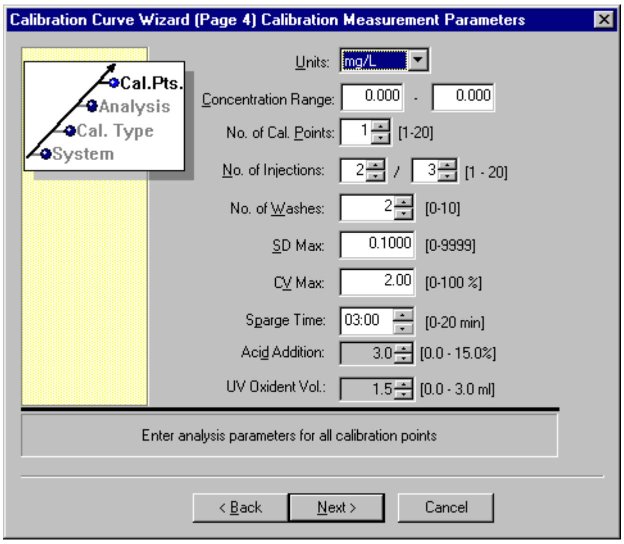


Figure 4.72 Calibration Curve Wizard (Page 4) Calibration Measurement Parameters

Parameter	Description
Units	Select the concentration units from the options in the drop-down list.
Concentration Range	Enter the maximum and minimum concentrations of the calibration curve standard solutions.
No. of Cal. Points	Enter the number of calibration standards that are used to create the calibration curve.
No. of Injections	Enter the minimum/maximum number of injections for the calibration standards.
No. of Washes	Specifies the number of times the syringe is washed with standard prior to the first injection. If the Auto Dilution option is selected, the standard is diluted and the syringe washed with the diluted standard. Enter an integer from 0 to 10.
SD Max and CV Max	Specifies the maximum acceptable standard deviation and coefficient of variation. Enter a value from 0 to 9999 for SD Max and from 0 to 100 for CV Max. If either of these values are met, no additional injections are required. If both of these values are exceeded, the calibration standards are automatically reinjected up to the maximum number of times. These fields are disabled if the No. of Injections is 1 or the minimum is equal to the maximum.
Sparge Time	Enter the length of time sparging will occur. (0 - 20 minutes)
Acid Addition	Enter the desired quantity of acid to be added. This option is available only for NPOC or related analyses.
UV Oxident Vol.	The oxidizer addition volume range is 0 - 3 mL. This is entered for TC or NPOC measurement.

Calibration Curve Wizard (Page 5) Calibration Points List

The Calibration Points List window displays a list of parameters for each calibration point. The parameters can be edited by using the buttons below the list. To edit calibration information a user must have access rights for updating data.

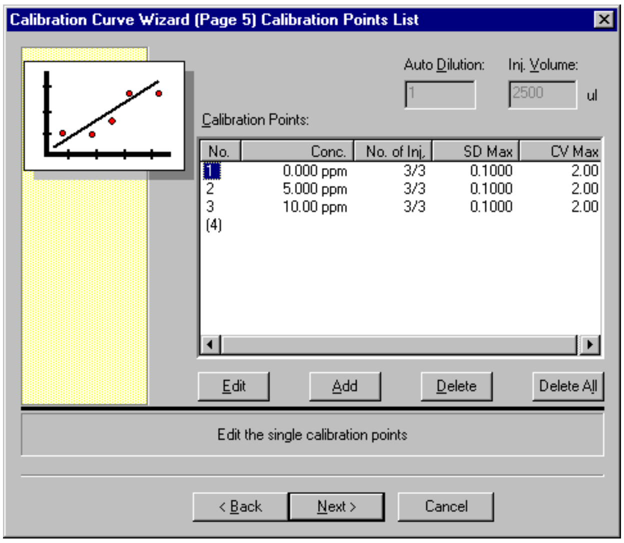


Figure 4.73 Calibration Curve Wizard (Page 5) Calibration Points List

Parameter	Description
Auto Dilution	The auto dilution factor is displayed. This field cannot be edited.
Inj. Volume	Displays the injection volume for the calibration standards. This field cannot be edited.
Calibration Points	Displays a table of measurement parameters (such as those entered in the previous page of the Calibration Curve Wizard) for each calibration point. To edit the parameters for a calibration point, highlight the point in the table and click the Edit button. To add a calibration point, click the appropriate number in the No. column to specify the insertion point, then click the Add button. To delete a calibration point, highlight the point in the table and click the Delete button. To delete all calibration points, click the Delete All button.

Edit Calibration Point Parameters

Click the Edit and Add buttons to display the Edit Calibration Point Parameters window. The window is used to enter calibration points manually or to edit the parameters of existing calibration points.

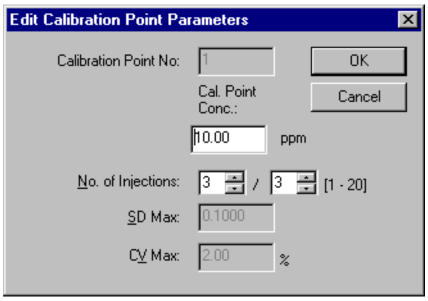


Figure 4.74 Edit Calibration Point Parameters Window

Parameter	Description
Calibration Point No.	Displays the calibration point number that was selected in the “No.” field of the Calibration Points List window. The number cannot be edited in this window.
Cal. Point Conc.	Displays the actual concentration of the calibration point. The concentration is calculated automatically based on the values entered into the Standard Solution Conc. and Auto Dilution fields.
No. of Injections	Enter the minimum and maximum number of injections.
SD Max and CV Max	Enter the maximum acceptable standard deviation or coefficient of variation values.

Calibration Curve Wizard (Page 6) Peak Time Parameters

The Peak Time Parameters window is used to set peak detection parameters.

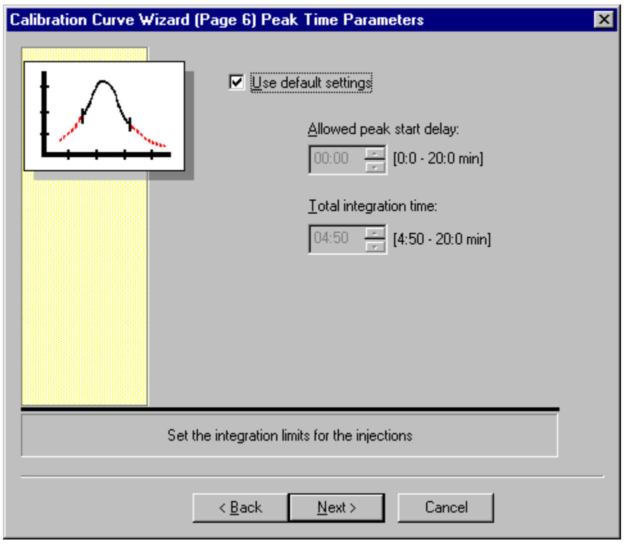


Figure 4.75 Calibration Curve Wizard (Page 6) Peak Time Parameters

Setting Item	Description
Use default settings	Select this option to use the default peak detection parameters. When this option is selected, all other options in this window are disabled.
Allowed peak start delay	The allowed peak start delay is the amount of time (in minutes) that can elapse before the analysis is stopped because a peak is not detected. Enter a number between 0 and 20:00.
Total integration time	The total integration time is the amount of time (in minutes) that analysis continues after the injection. If the peak end is determined prior to the set time, integration stops and the next injection is made. Enter a number between 4:50 and 20:00.

Calibration Curve Wizard (Page 7) History

The History window is used to enable the History Log. The History Log records all modifications to the calibration curve parameters. The History Log can be set to require users to enter comments for each modification. If the History Log is not required, it need not be enabled, as the software operates properly without it.

Note: Once the History Log is enabled, it cannot be edited. All modifications to the calibration curve parameters are tracked. If the mandatory comment option is selected, the user is required to enter a comment each time any parameter in the calibration curve is modified.

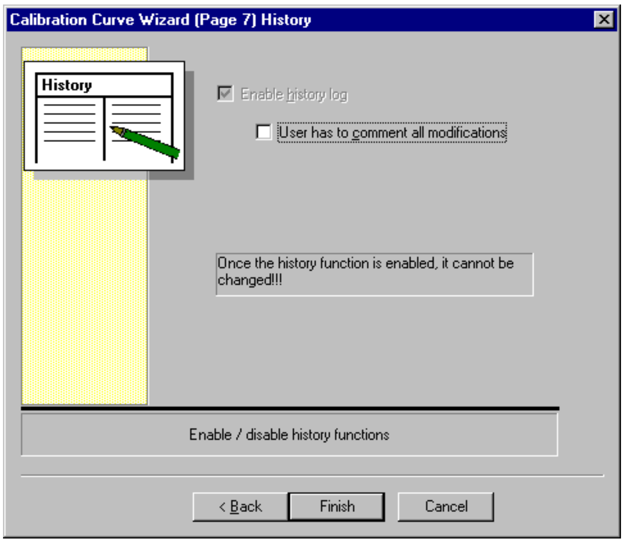


Figure 4.76 Calibration Curve Wizard (Page 7) History

Setting Item	Description
Enable history log	Select this option to enable the software to monitor all modifications to the system.
User has to comment all modifications	Select this option to require the user to add a comment to all modifications.

4.4.2.3 Method Wizard

The Method Wizard is used to set method parameters and is accessed by selecting File>New>Method. The Method Wizard consists of several windows, each of which is described below. The Back and Next buttons are used to navigate between windows. Each set of parameters selected for a specific method applies only to that method.

Note: Selected windows in the Method Wizard are repeated for combined analysis types such as TOC. Each duplicate window represents one analysis type. Parameters that are not applicable to an analysis type will not appear in the Method Wizard window.

Method Wizard (Page 1) System Information

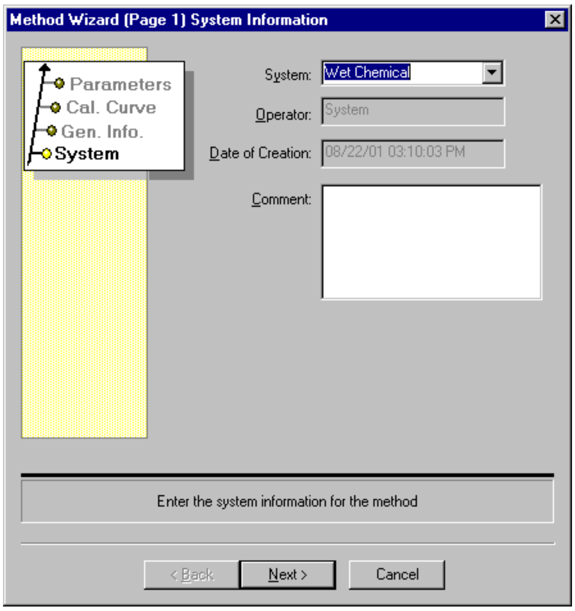


Figure 4.77 Method Wizard (Page 1) System Information

Setting Item	Description
System	Select one of the configured instruments.
Operator	Displays the current operator name.
Date of Creation	Displays the current time.
Comment	Enter a comment (512 characters maximum), if desired.

Method Wizard (Page 2) Analysis Information

The Analysis Information window is used to set analysis parameters for the method being created.

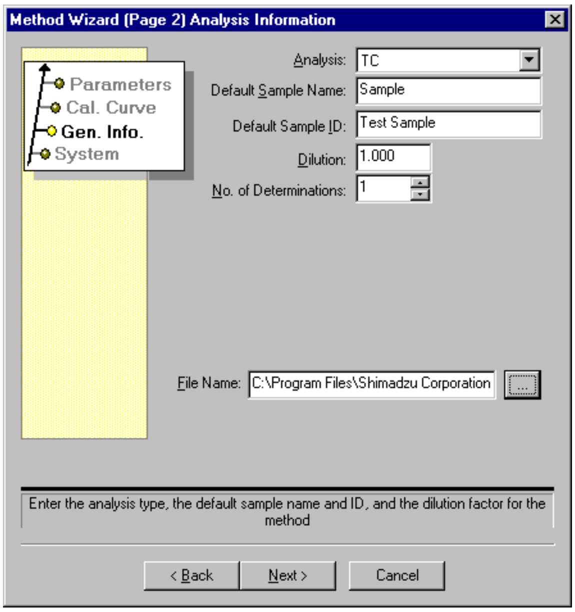


Figure 4.78 Method Wizard (Page 2) Analysis Information

Setting Item	Description
Analysis	Select the analysis type from the drop-down list.
Default Sample Name	Enter the default sample name (64 characters maximum).
Default Sample ID	Enter the default sample ID (64 characters maximum).
Dilution	Enter the preparation dilution factor, which is used to calculate concentration results. The default value is 1.
No. of Determinations	Enter the number of times each sample is measured. Determinations and injections are different; each determination is made up of one or more injections.
File Name	Enter the name of the new method. Click the Browse button to save the file in a directory other than the default directory.

Method Wizard (Page 3) Calibration Curves

The Calibration Curves window is used to specify the calibration curves used to calculate results for samples analyzed by the method. Up to 3 calibration curves can be entered and, during analysis, the system automatically determines which curve is most suitable for the sample. Refer to Section 6.2.4 "Automatic Selection of the Optimal Calibration Curve" for more details.

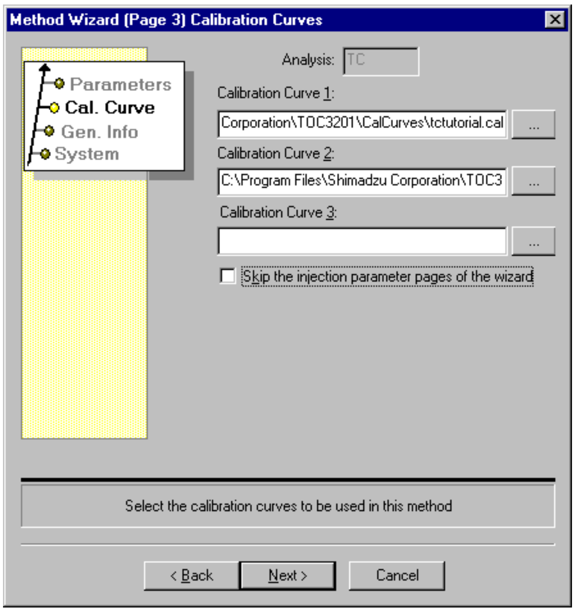


Figure 4.79 Method Wizard (Page 3) Calibration Curves

Setting Item	Description
Analysis	Displays the previously selected analysis type.
Calibration Curve 1/2/3	Enter the name of the calibration curve or click the Browse button to select a file using the File>Open dialog box.
Skip the injection parameter pages of the wizard	Select this item to skip the Injection Parameters window of the Method Wizard. Use this option when the sample injection parameters are the same as those used for calibration curve measurements.

Method Wizard (Page 4) Injection Parameters

The Injection Parameters window is used to specify the injection parameters for the method.

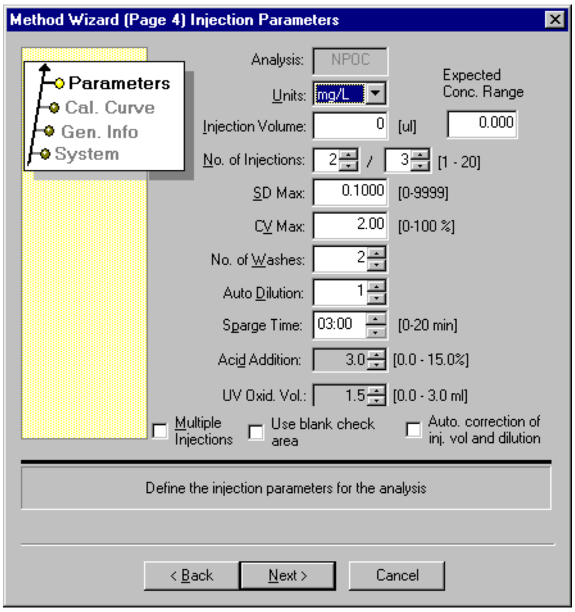


Figure 4.80 Method Wizard (Page 4) Injection Parameters

Setting Item	Description
Analysis	Displays the previously selected analysis type.
Units	Select the concentration units to be used from the drop down list.
Injection Volume	Enter the injection volume.
Expected Conc. Range	Enter the expected sample concentration if it is known.
No. of Injections	Enter the minimum/maximum number of injections for the samples.
SD Max	Enter the maximum acceptable standard deviation. If this value is met, no additional injections are required. However, if both this value and the CV Max value are exceeded, the samples are automatically reinjected up to the maximum number of times. The SD Max field is disabled if the No. of Injections is 1 or if the minimum is equal to the maximum.
CV Max	Enter the maximum acceptable coefficient of variation. If this value is met, no additional injections are required. However, if both this value and the SD Max value are exceeded, the samples are automatically reinjected up to the maximum number of times. The CV Max field is disabled if the No. of Injections is 1 or if the maximum and minimum values are the same.
No. of Washes	Enter the number of times the syringe is washed with sample prior to the first injection. If the Auto Dilution option was selected, the sample is diluted and the syringe washed with the diluted sample.
Auto Dilution	Enter a dilution factor for the samples. The instrument automatically dilutes the samples by this factor, and this factor is used to calculate the final result.

Setting Item	Description
Sparge Time	Enter the desired sparge time. This option is available only for NPOC.
Acid Addition	Enter the desired quantity of acid to be added.
UV Oxid. Vol.	Sets the volume of oxidizer solution to be added to the sample.
Use blank check area	Select to include blank check result in the concentration calculation.
Auto correction of inj. vol. and dilution	Select to enable the instrument to automatically adjust the injection volume and dilution factor when the peak height for the initial injection exceeds full scale. After the adjustment, measurement is repeated.

Method Wizard (Page 5) Peak Time Parameters

The Peak Time Parameters window is used to set peak integration parameters.

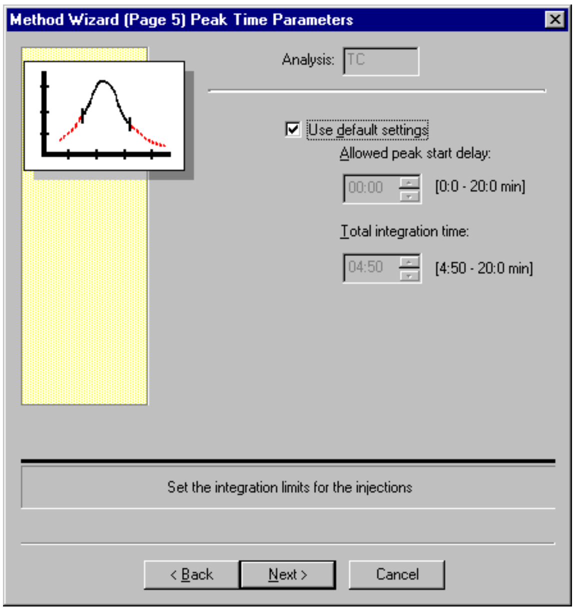


Figure 4.81 Method Wizard (Page 5) Peak Time Parameters

Setting Item	Description
Analysis	Displays the previously selected analysis type.
Use default settings	Select this option to use the default integration parameters. When this option is selected, all other options in this window are disabled.
Allowed peak start delay	The allowed peak start delay is the amount of time (in minutes) that can elapse before the analysis is stopped because a peak is not detected. Enter a number between 0 and 20:00.
Total integration time	The total integration time is the amount of time (in minutes) that analysis continues after the injection. If the peak end is determined prior to the set time, integration stops and the next injection is made. Enter a number between 4:50 and 20:00.

Method Wizard (Page 6) USP/EP

The USP/EP window is displayed only for TC or NPOC methods, and is used to allow samples to be checked in accordance with USP standards. Place a check in the box to enable this function.

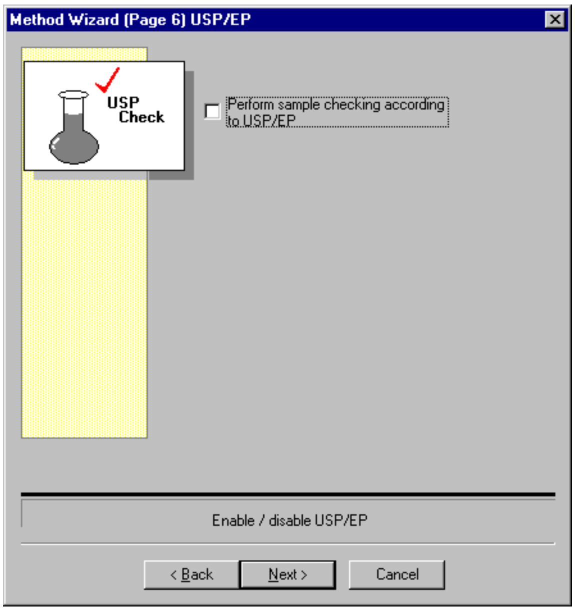


Figure 4.82 Method Wizard (Page 6) USP/EP

Method Wizard (Page 7) History

The History window is used to enable the History Log. The History Log records all modifications to the method. The History Log can be set to require users to enter comments for each modification.

Note: Once the History function is enabled, it cannot be disabled. All modifications to the method are tracked. If the mandatory comment option is selected, the user is required to enter a comment each time any method parameter is modified.

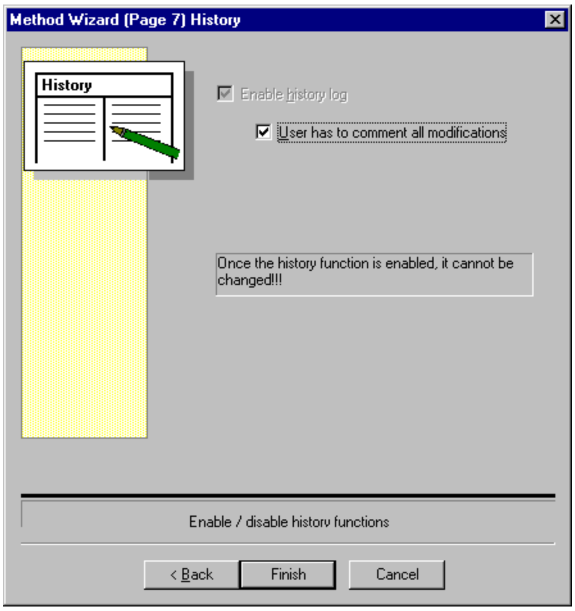


Figure 4.83 Method Wizard (Page 7) History

Setting Item	Description
Enable history log	Select this option to enable the software to monitor all modifications to the system. Once the History Log is enabled, it cannot be changed.
User has to comment all modifications	Select this option to require comments or approval for all method modifications. If the History Log is not required, it need not be enabled, as the software operates properly without it.

4.4.2.4 Open

The Open command is used to select and open a Sample Table, Method, Calibration Curve, Parameter, or Control Sample Template file. Save the Sample Table before executing the Open command. More than one Sample Table can be opened at the same time. Select the Open command to display the Open dialog box.

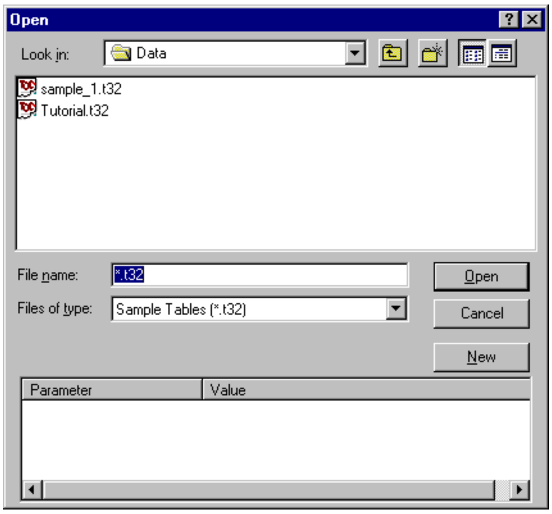


Figure 4.84 Open Dialog Box

Table 4.5 “Acceptable File Types in the Open Function” displays the types of files that can be opened using this function. No other file types should be opened using this menu command.

Table 4.5 Acceptable File Types in the Open Function

File Extension	Description
.t32	Sample Table
.met	Method
.cal	Calibration Curve
.tpl	Control Sample Template
.trd	Data Profile

The opened file may replace the currently displayed table or function, depending on the type of file selected.

Dialog Box Options	Description
Look in	Find the file to be opened by choosing the appropriate folder or accessing other directories. The selected file will appear in the File name text box.
File name	Enter the name of the file to be opened.
Files of type	Select the type of file from the drop-down list. Choose only files of type *.t32 when opening a Sample Table.
Open button	Opens the selected file.
Cancel button	Aborts the Open operation.

4.4.2.5 Close

The Close command is used to close the current window and is an option only when a file is open. If the open file has not been saved and the Close command is selected, the Save As dialog box is displayed. Refer to Section 4.4.2.7 "Save As" for details. Enter a name for the file and click the OK button to save the file. If the open file has been modified but not saved and the Close command is selected, a dialog box is displayed as shown below. Click the Yes button to save the file before closing. Click the No button to close the file without saving the modified information. Click the Cancel button to abort the Close command and return to the file.

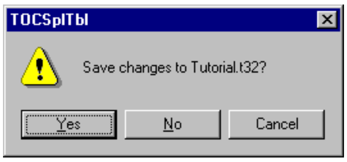


Figure 4.85 Dialog Box for Saving Modified Data

4.4.2.6 Save

The Save command is used to save data in the current active file. If the file has not yet been saved and the Save command is selected, the Save As dialog box is displayed. Refer to Section 4.4.2.7 "Save As" for details.

4.4.2.7 Save As

The Save As command is used to save the current active file with a new file name. Selecting the command opens the Save As dialog box shown below. Button options in the dialog box are similar to those in the Open dialog box. Refer to Section 4.4.2.4 "Open" for more details. Click the Save button to save the data under the specified file name. Click the Cancel button to abort the Save As operation.

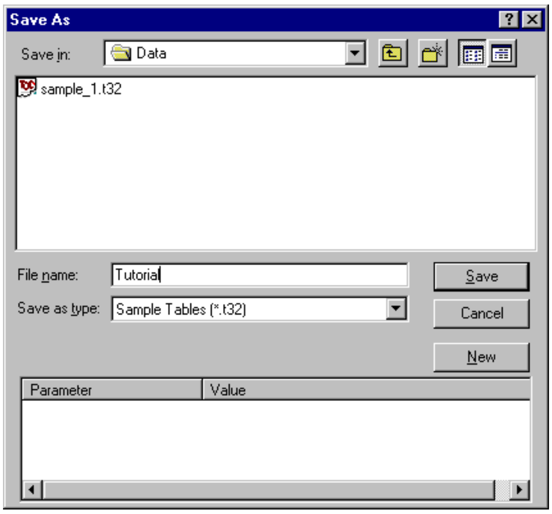


Figure 4.86 Save As Dialog Box

Dialog Box Options	Description
Save in	Select the directory in which the file is to be saved by choosing the folder or accessing other directories from the drop-down list. The currently selected directory appears in the Save in text field.
File name	Enter a name for the file. The applicable file extension will automatically be added if it is not entered.
Save as type	Select the type of file from the drop-down list. A list of available file types is listed in Table 4.2.

4.4.2.8 ASCII Export Options

The ASCII Export function is used to export Sample Table data to an ASCII file. Open a Sample Table that contains finished measurement data. Select ASCII Export from the File menu. The Save As dialog box is displayed. Refer to Section 4.4.2.7 "Save As" for details. Enter a file name for the ASCII file to be created and confirm that the "Save as type" field contains the ASCII file type (*.txt). If the entered file name matches a file that already exists, a confirmation message is displayed.

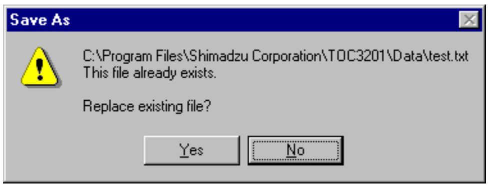


Figure 4.87 Save As Confirmation Message

Click the Yes button to overwrite the existing file and close the dialog box. Click the No button to redisplay the Save As dialog box and enter a new file name.

Select the ASCII Export Options command from the File menu. The ASCII Export Options dialog box, which contains several tabs, is displayed. Each tab contains a list of export options, as shown in the figures below. On each tab, certain items are selected by default. Individual options can be selected or deselected by clicking in the check box. Use the Select All and Select None buttons to quickly select and deselect all options on a tab. Use the Default button to select the default options on a tab. Click the Cancel button to exit the ASCII Export Options dialog box without saving changes. Click the OK button to save changes and exit the ASCII Export Options window.

Header Tab

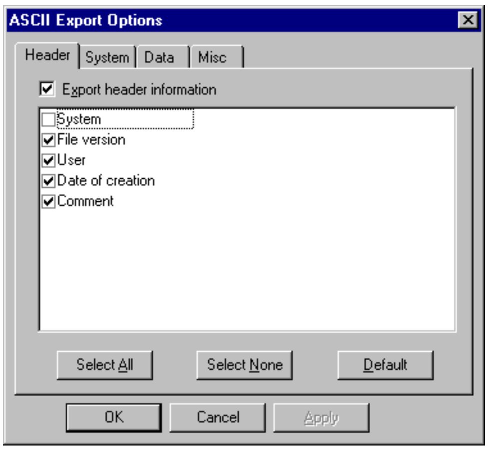


Figure 4.88 ASCII Export Options Window: Header Tab

Setting Item	Description
Export header information	Select to enable export of the selected header options. This option is selected by default.

System Tab

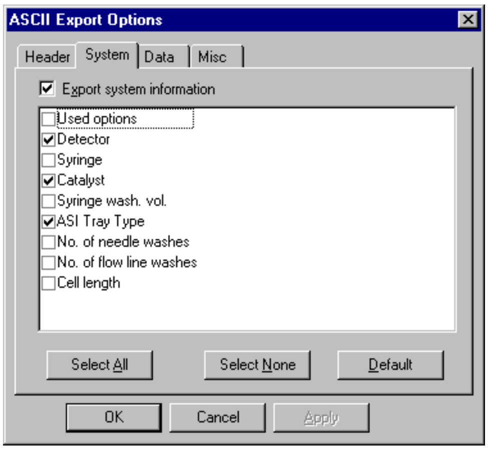


Figure 4.89 ASCII Export Options Window: System Tab

Setting Item	Description
Export system information	Select to enable export of the selected system options. This option is selected by default.

Data Tab

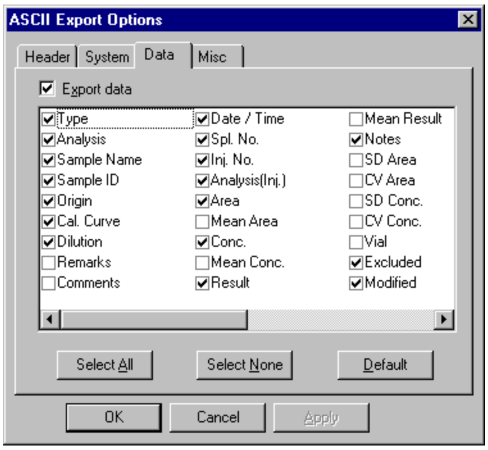


Figure 4.90 ASCII Export Options Window: Data Tab

Setting Item	Description
Export data	Select to enable export of the selected data options. This option is selected by default.

Miscellaneous Tab

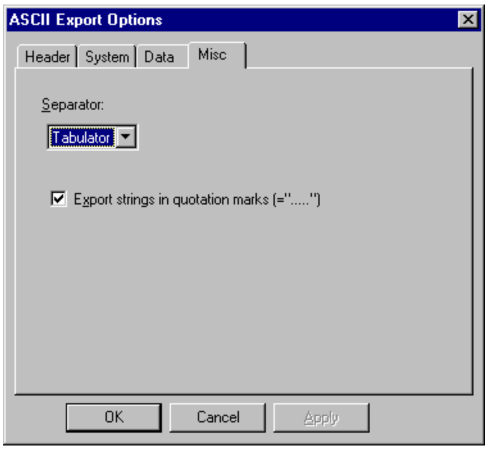


Figure 4.91 ASCII Export Options Window: Miscellaneous Tab

The Miscellaneous tab contains additional options for the format of exported data. To change data format, open a Sample Table, select File>ASCII Export Options, then select the file to which changes should be made. On the Miscellaneous tab, select the desired type of Separator from the drop-down list. The Separator is the type of delimiter used to separate items of data. Select the “Export strings in quotation marks” option to enclose all of the character string items in quotation marks.

4.4.2.9 Database Export

The Database Export command is used to enable automatic export of data to an external (Microsoft® Access) database during analysis. Selecting the command displays the Database Options window.

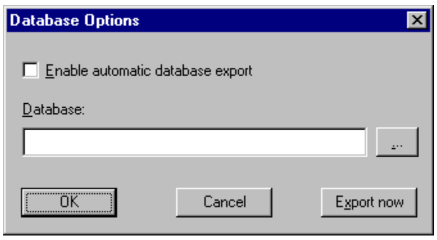


Figure 4.92 Database Options Window

Setting Item	Description
Enable automatic database export	Select this option to enable the database export function
Database	Enter the name and file path of the database, or click the Browse button to select the database using the File>Open dialog box.

4.4.2.10 Print>Table

The Print>Table command is used to print the active Sample Table in a table format. Select this command to open the Print dialog box, which may have a variable display depending on the installed printer driver. Specify items to be included in the printout by using the Page Setup command. Refer to Section 4.4.2.15 “Page Setup” for more details.

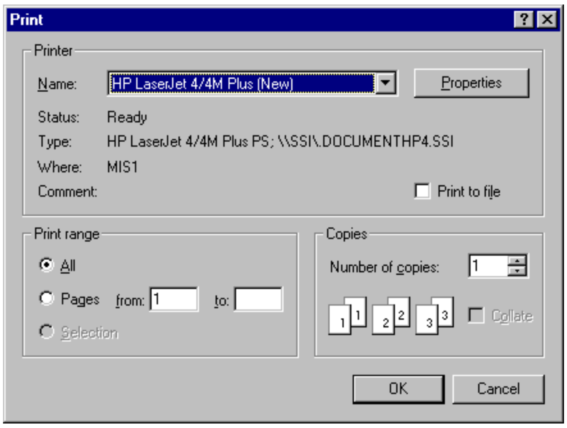


Figure 4.93 Print Dialog Box

The Name text field identifies the printer used to print the table. Specify a different printer by selecting it from the drop-down list.

Setting Item	Description
Properties	Modify the printer settings using standard Windows procedures.
Print range	Click the All radio button to print all pages of the table. Click the Pages radio button and enter the page numbers to be printed if all pages are not desired.
Number of copies	Enter the number of copies to be printed.

4.4.2.11 Print>Sample Report

The Print>Sample Report command is used to open the Print dialog box to print the Sample Table in report format. The print dialog box is the same as that described in Section 4.4.2.10 “Print>Table”.

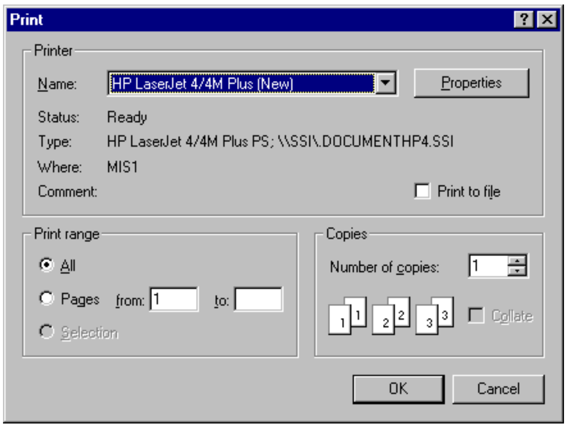


Figure 4.94 Print Dialog Box

4.4.2.12 Print Preview>Table

The Print Preview>Table option is used to display the sample table before printing. Use the buttons at the top of the Print Preview window to change the display or print the table, as described below.

Button	Description
Next Page and Previous Page	Click the Next Page button to display the next page of the report. Click the Previous page button to display the previous page of the report. The Next Page button is disabled when the last page of the report is displayed, and the Previous Page button is disabled when the first page of the report is displayed.
One Page/Two Page	Click the One Page button to display one page at a time in the Print Preview window. After selection, the button toggles to the Two Page button. Click the Two Page button to display two pages side by side.
Zoom In and Zoom Out	Click the Zoom In and Zoom Out buttons to change the magnification of the displayed pages. Three different zoom factors are available. Repeated selection of either option will continue to increase or decrease the magnification, as applicable.
Close	Click the Close button to close the Print Preview window and return to the previous window. The Print Preview window must be closed to edit the report.
Print	Click the Print button to print the report.

4.4.2.13 Print Preview>Sample Report

The Print Preview>Sample Report option is used to display the sample report before printing. Use the buttons at the top of the Print Preview window to change the display or print the report, as described in Section 4.4.2.12 "Print Preview>Table".

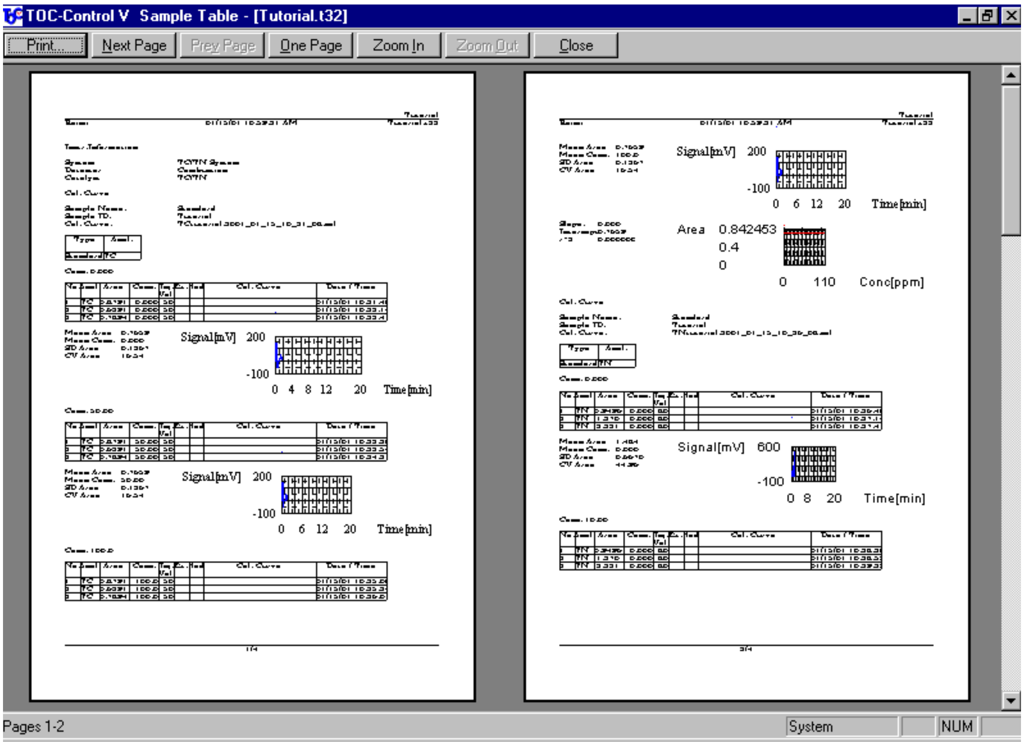


Figure 4.95 Print Preview>Report Window

4.4.2.14 Print Setup

The Print Setup command is used to set print options. Select this command to open the Print Setup dialog box, which may have a variable display depending on the installed printer driver. Modifications to the Print Setup dialog box are saved until the TOC-Control V software is exited.

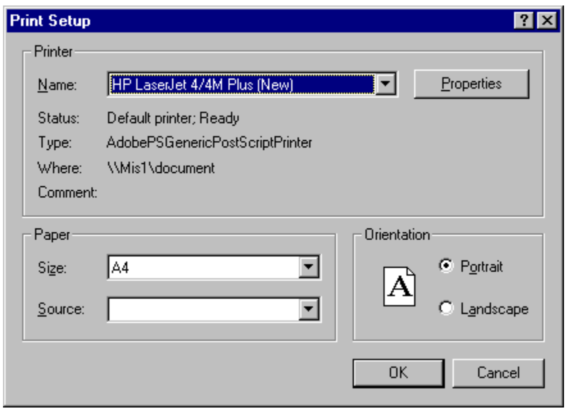


Figure 4.96 Print Setup Dialog Box

4.4.2.15 Page Setup

The Page Setup command is used to select items to be printed on a report or table and to specify format parameters such as margins, headers, and footers. Selecting the command displays the Page Setup window, which consists of several tabs. Each tab contains a list of page setup options shown in the figures below. On each tab, certain options are selected by default. Individual options can be selected or deselected by clicking the check box. Use the Select All and Select None buttons to quickly select and deselect all options on a tab. Use the Default button to select the default options for a tab. Click the Cancel button to exit the Page Setup window without saving changes. Click the OK button to save changes and exit the Page Setup window.

Instrument Tab

The Instrument tab displays a list of instrument information items that can be printed on the report.

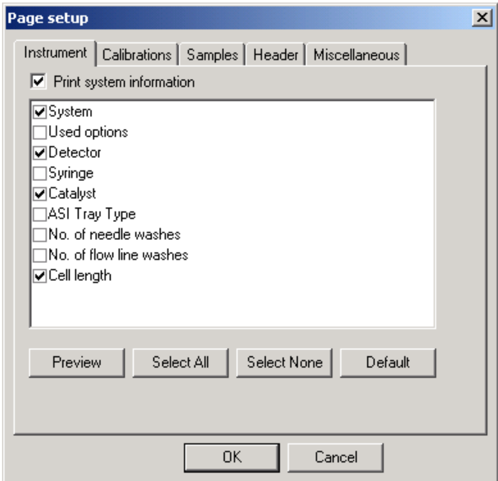


Figure 4.97 Page Setup Window: Instrument Tab

Calibrations Tab

The Calibrations tab displays a list of calibration information items that can be printed on the report.

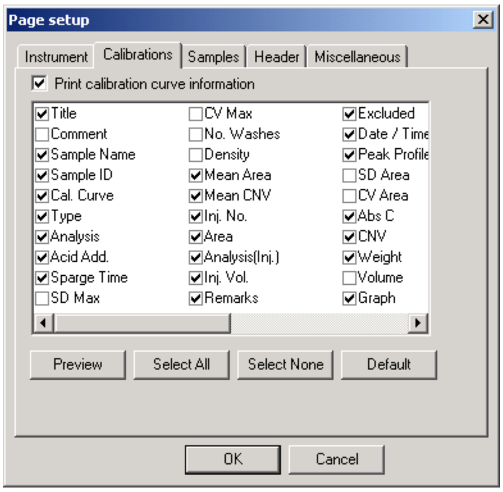


Figure 4.98 Page Setup Window: Calibrations Tab

Samples Tab

The Samples tab displays a list of sample information items that can be printed on the report.

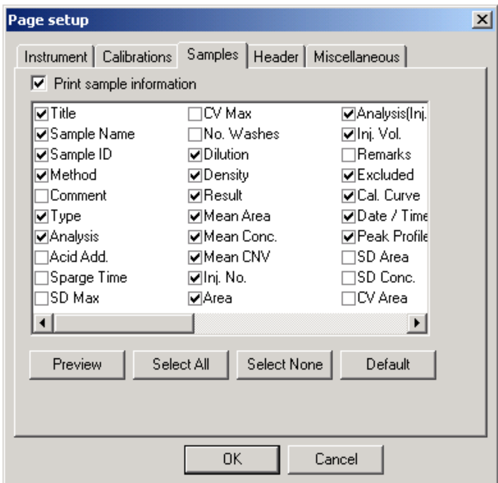


Figure 4.99 Page Setup Window: Samples Tab

Header Tab

The Header tab displays a list of header items that can be printed on the report.

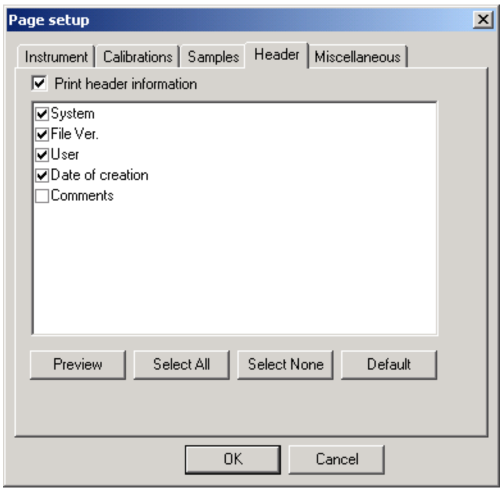


Figure 4.100 Page Setup Window: Header Tab

Miscellaneous Tab

The Miscellaneous tab displays a list of format items for the report, as described below.

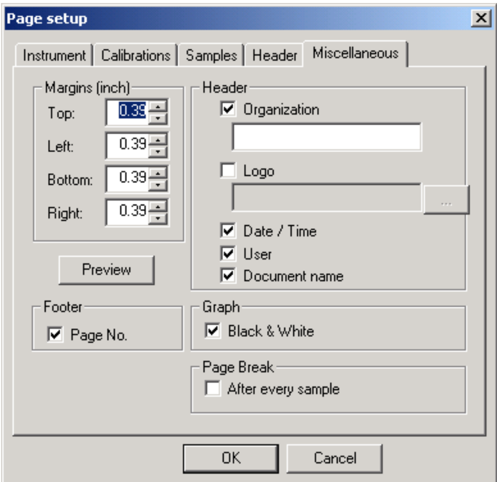


Figure 4.101 Page Setup Window: Miscellaneous Tab

Setting Item	Description
Margins	Use the spin controls to enter the page margins (in mm).
Footer, Page No.	Select this option to print the page number in the footer of each page.
Header	*Organization:* Select this option and enter the name of the organization in the text box to print the organization name in the page header. *Logo:* Select this option to print a bitmap in the page header. Use the Browse button to select the bitmap file. *Date/Time:* Select this option to print the current system date and time in the page header. *User:* Select this option to print the current user name in the page header. *Document name:* Select this option to print the document file name in the page header.

4.4.2.16 Exit

The Exit command is used to exit the Sample Table Editor. If the active file has been modified but not saved and the Exit command is selected, a dialog box is displayed as shown below. Click the Yes button to save the file before exiting. Click the No button to exit without saving the modified information. Click the Cancel button to abort the Exit command and return to the active file.

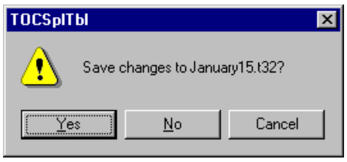


Figure 4.102 Save Changes Prompt

If the software is connected to an instrument and the Exit command is selected, a dialog box is displayed as shown below. Click the OK button to disconnect the instrument before exiting the software. Click the Cancel button to abort the Exit command.

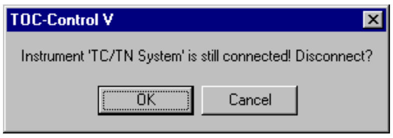


Figure 4.103 Disconnect Instrument Prompt

4.4.3 Edit Menu

The Edit menu contains functions that allow a user to modify data. To change data using the Edit menu functions, a user must have the appropriate access rights. Options such as Cut, Copy, and Paste are available only when a content selection from an open file is made.

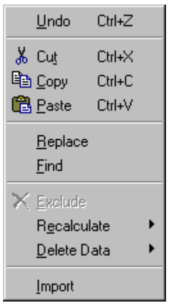


Figure 4.104 Edit Menu

4.4.3.1 Undo

The Undo function is used to reverse the last change made in the active Sample Table and is only available after the user changes the content of a cell in the table. The Undo function is not available during actual measurement or when the user has inserted or deleted finished samples, calibration curves, or control samples.

4.4.3.2 Cut/Copy/Paste

The Cut, Copy, and Paste functions are used to edit selected Sample Table cells and rows. The Cut command deletes selected information from rows or cells and stores it on the clipboard. The Cut command cannot be used to delete entire samples after measurement is complete. The Copy command copies selected data from rows or cells and stores it on the clipboard. The Paste command copies the contents of the clipboard to the selected cells or rows of the table. When pasting an entire row, click the destination row number to highlight the entire destination row, then execute the Paste command.

Note: When pasting, do not select a destination row that currently contains data. The data will be overwritten!

- The Cut, Copy, and Paste commands can also be accessed by clicking the icons on the Toolbar.

4.4.3.3 Replace

The Replace function is used to replace text in Sample Table cells with specified text. Select the Replace function to open the Replace dialog box. The Replace command can be used to replace data in the Sample Name, Sample ID, Dilution, and Comment fields of the Sample Table. The command cannot be used to replace data for samples after measurement is complete.

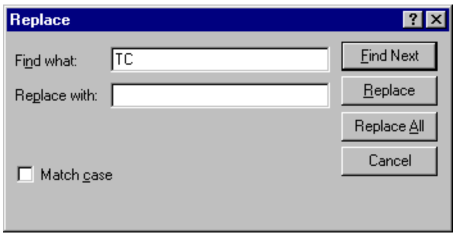


Figure 4.105 Replace Dialog Box

Setting Item	Description
Find what	Enter the text to be searched for.
Replace with	Enter the text that the searched-for text should be replaced with.
Match case	Select to perform a case-sensitive text search.
Find Next	Select to begin the search or to continue a search that has already begun.
Replace	Select to replace the found text with the text in the "Replace with" field.
Replace All	Select to replace all occurrences of found text with the text in the "Replace with" field.
Cancel	Select to close the Replace dialog box.

4.4.3.4 Find

The Find function is used to search for specified text. Select the Find command to open the Find dialog box, which is similar to the Replace dialog box and displays the options described below. Refer to Section 4.4.3.3 "Replace" for more details.

Setting Item	Description
Find what	Enter the text to be searched for.
Match case	Select to perform a case-sensitive text search.
Direction	Click the Up radio button to search the table backwards from the current location. Click the Down radio button to search the table forward from the current location.
Find Next	Select to begin the search or to continue a search that has already begun.
Cancel	Select to close the Find dialog box.

4.4.3.5 Exclude

The Exclude function is used to exclude selected injections from the calculations. A user must have access rights for updating data to use the Exclude function. To exclude data, highlight the desired injection shown in the Sample Window Injection Table and select Exclude from the Edit menu. A confirmation dialog box is displayed. Click the Yes button to exclude the selected data. Click the No button to abort the operation.

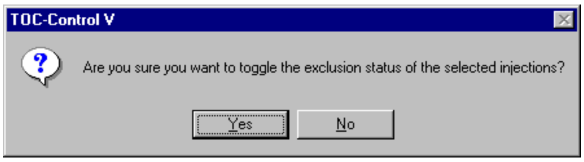


Figure 4.106 Exclude Confirmation Dialog Box

4.4.3.6 Recalculate>All

The Recalculate>All function is used to recalculate the data for all measured samples in a Sample Table after applying a new calibration curve or excluding injections. To execute the command, open the appropriate Sample Table and select Recalculate All from the Edit menu. A message is displayed in the Notify folder of the Notification bar when the recalculation is complete.

4.4.3.7 Recalculate>Highlighted

The Recalculate>Highlighted function is used to recalculate the data for selected samples after applying a new calibration curve or excluding injections. The function is similar to the Recalculate All function described in Section 4.4.3.6 “Recalculate>All” of this manual. To execute the command, open the appropriate Sample Table, highlight the samples to be recalculated, and select Recalculate Highlighted from the Edit menu.

4.4.3.8 Delete Data>All

The Delete Data>All function can only be used if the GLP/GMP conformity was disabled during system installation. The function is used to delete all measurement data in the Sample Table. The Sample Table structure can then be used to enter new data. A user must have access rights for updating data to use the Delete Data>All function.

To use the Delete Data>All function, open a Sample Table that contains measurement data and select Delete Data>All from the Edit menu. A confirmation dialog box is displayed, as shown in the figure below. Click the Yes button to delete the data. Click the No button to abort the operation. A message is displayed in the Notification bar when the data has been deleted.

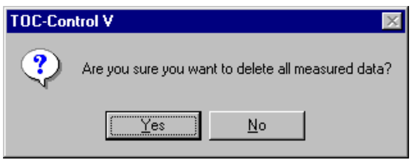


Figure 4.107 Delete Data Confirmation Dialog Box

4.4.3.9 Delete Data>Highlighted

The Delete Data>Highlighted function is used to delete the measurement data for selected samples. To execute the command, open the appropriate Sample Table, highlight the samples to be deleted, and select Delete Data>Highlighted from the Edit menu. A message is displayed on the Notifications tab in the Notification bar when the selected data has been deleted.

4.4.3.10 Import

The Import function is used to import data from an ASCII file into a Sample Table. The Import function is not available during sample analysis. To import data, open the destination Sample Table and place the cursor in the row of the table where the data will be imported. Select the Import function from the Edit menu. The Open dialog box is displayed. Select the ASCII file from where the data will be imported. The data is inserted in the current position of the active Sample Table.

4.4.4 View Menu

The View menu contains options for viewing calibration, method, and sample information, and for selecting settings for the display screen.

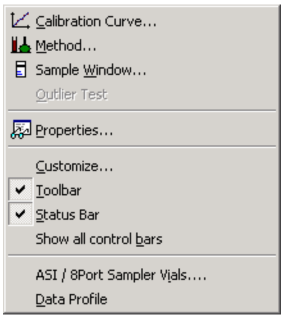


Figure 4.108 View Menu

4.4.4.1 Calibration Curve

The View>Calibration Curve command is used to view the calibration curve information associated with a sample. Place the cursor in the desired row of the Injection Table and select the Calibration Curve command from the View menu. The Calibration Curve Properties window is displayed. Refer to Section 4.4.4.5 “Properties” for more details.

TIP: *The Calibration Curve Properties window can also be displayed by clicking the Calibration Curve (View) button on the Toolbar.*

4.4.4.2 Method

The View>Method command is used to view the properties associated with a method. Selecting the command displays the Sample/Method Properties window, which consists of several tabs, including a separate tab for each applicable method. Each tab lists options for method properties, as shown in the figures below. Individual options can be selected, deselected, or edited by clicking in the check boxes or entering information into the text fields. Certain options are disabled if sample analysis is complete. Each tab contains the same command buttons: OK, Cancel and Apply. Click the OK button to save changes and exit the Sample/Method Properties window. Click the Cancel button to exit the Sample/Method Properties window without saving changes. Click the Apply button to save changes without exiting the Sample/Method properties window.

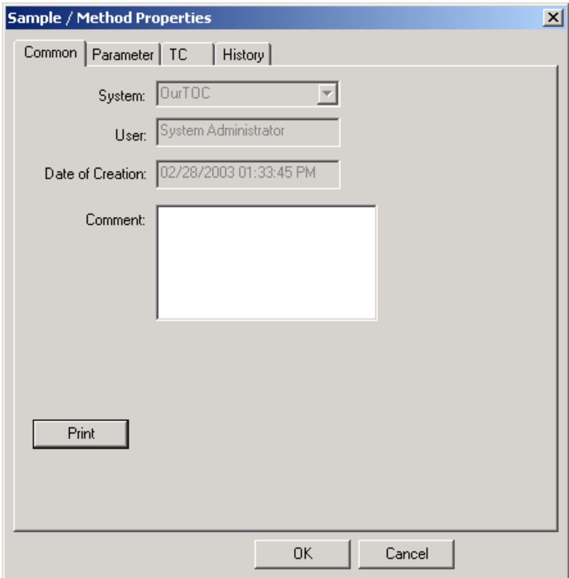


Figure 4.109 Sample/Method Properties Window: Common Tab

Common Tab

Setting Item	Description
System	Displays the system currently in use.
User	Displays the name of the user who created the method.
Date of Creation	Displays the date on which the method was created.
Comment	Enter a comment (512 characters maximum).
Print	Prints the calibration curve information

Parameter Tab

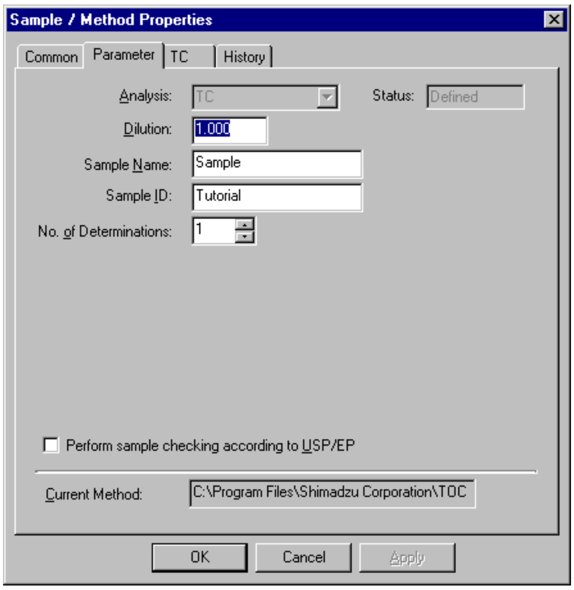


Figure 4.110 Sample/Method Properties Window: Parameter Tab

Setting Item	Description
Analysis	Displays analysis type. Changes cannot be made to this field.
Dilution	Displays the preparation dilution factor, which is used to calculate sample concentrations. This field can be edited.
Sample Name	Displays the default sample name (64 characters maximum). This field can be edited.
Sample ID	Displays the default sample ID (64 characters maximum). This field can be edited.
No. of Determinations	Displays the number of times (1-10) the sample is measured. Determinations and injections are different; each determination is made up of one or more injections. This field can be edited.
Perform sample checking according to USP/EP	Select to enable USP sample checking.
Current Method	Displays the file name of the current method. Changes cannot be made to this field.

TC Tab

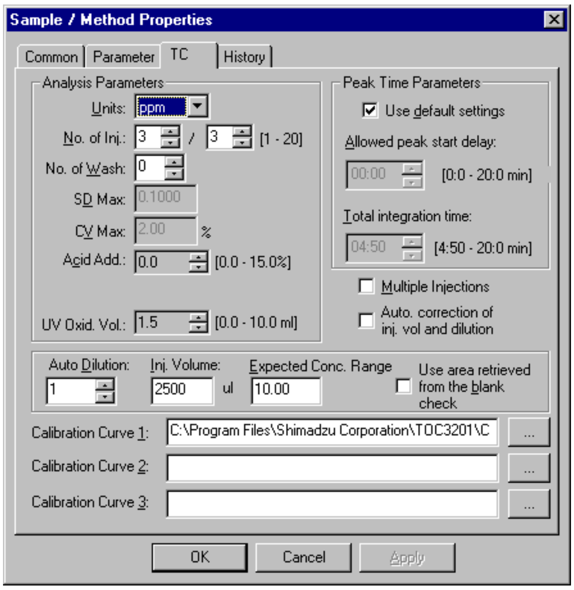


Figure 4.111 Sample/Method Properties Window: TC Tab

Analysis Parameters

Setting Item	Description
Units	Displays the concentration units. This field can be edited by selecting another option from the drop-down list.
No. of Inj.	Displays the minimum/maximum number of injections for the samples analyzed with this method. This field can be edited.
No. of Wash	Displays the number of times the syringe is washed with sample prior to the first injection. If the Auto Dilution option was selected, the sample is diluted and the syringe washed with the diluted sample. This field can be edited.
SD Max	Displays the maximum acceptable standard deviation. If this value is met, no additional injections are required. However, if both this value and the CV Max value are exceeded, the samples are automatically reinjected up to the maximum number of times. This field can be edited.
CV Max	Displays the maximum acceptable coefficient of variation. If this value is met, no additional injections are required. However, if both this value and the SD Max value are exceeded, the samples are automatically reinjected up to the maximum number of times. This field can be edited.
Acid Add	Displays the quantity of acid to be added to each sample. This value is expressed as a percent of the sample volume. This option is available only for NPOC and related analyses. This field can be edited.
UV Oxid Vol.	Enter the volume of oxidizer solution to be used.

Peak Time Parameters

Setting Item	Description
Use default settings	Select this option to use the default settings for peak integration.
Allowed peak start delay	Displays the amount of time (in minutes) that can elapse before the analysis is stopped because a peak is not detected. This field can be edited by entering a number between 0 and 20:00. This option is disabled when *Use default settings* is selected.
Total integration time	The total integration time is the amount of time (in minutes) that analysis continues after the the injection. If the peak end is determined prior to the set time, integration stops and the next injection is made. Enter a number between 4:50 and 20:00.This option is disabled when *Use default settings* is selected.

Other Parameters

Setting Item	Description
Auto correction of inj. volume and dilution	Select this option to enable the instrument to automatically adjust the injection volume and dilution factor when the peak height for the initial injection exceeds full scale. After the adjustment, measurement is repeated.
Auto Dilution	Displays the factor by which the instrument will automatically dilute the sample. This field can be edited.
Inj. Volume	Displays the injection volume. This field can be edited.
Expected Conc. Range	Enter the expected concentration if it is known.
Use area retrieved from the blank check	Select this option to include the blank check result in the concentration calculation.
Calibration Curve 1/2/3	Displays the name(s) of calibration curve(s) used to quantitate samples analyzed by the method. These fields can be edited by entering a calibration curve file name or clicking the Browse button to search for the file.

IC Tab

The IC tab options are similar to those described above for the TC tab, except that the Acid Add and UV Oxid. Vol. parameters are not available.

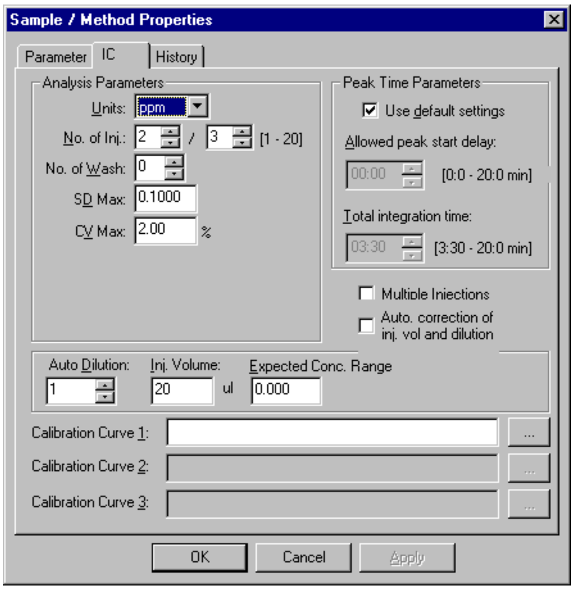


Figure 4.112 Sample/Method Properties Window: IC Tab

NPOC Tab

The NPOC tab options are similar to those described above for the TC tab.

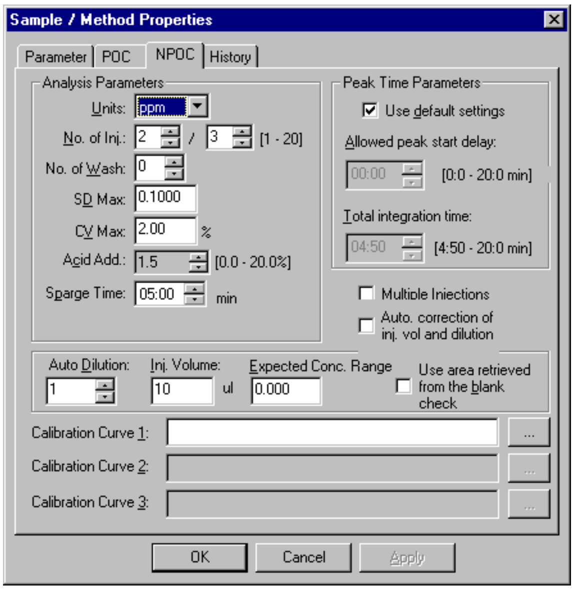


Figure 4.113 Sample/Method Properties Window: NPOC Tab

History Tab

If the History Log was enabled when the method was created, the History tab displays a list of changes made to the method parameters. If the History Log was not enabled previously, it can be enabled on the History tab.

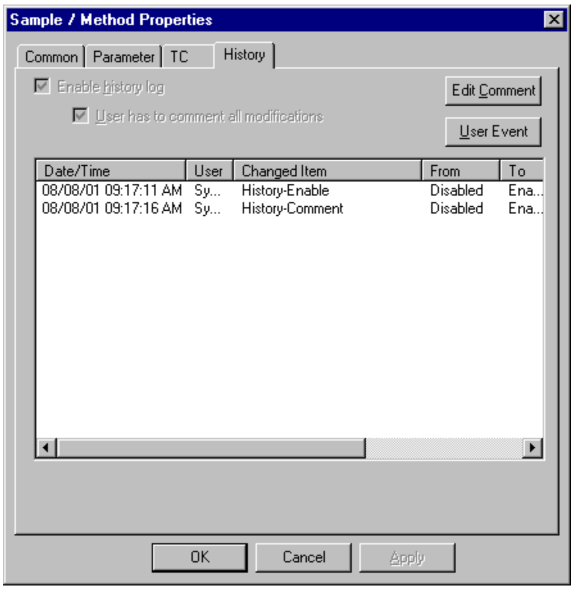


Figure 4.114 Sample/Method Properties Window: History Tab

Setting Item	Description
Enable history log	Select this option to enable the software to monitor all modifications to the system. If the History Log is not required, it need not be enabled, as the software operates properly without it. Once the History log is enabled, it cannot be disabled.
User has to comment all modifications	Select this option to require comments or approval for all modifications. If the History Log is not required, it need not be enabled, as the software operates properly without it.
ASCII Export	The displayed items in the list are exported as a tab-delimited text file.

A list of the Comment dialog box contents is shown below:

Table 4.6 Comment Dialog Box Contents

Item	Description
Date/Time	Displays the date and time of the modification.
User	Displays the user who made the modification.
Changed item	Describes the parameter that was changed.
From	Displays the previous parameter value.
To	Displays the new parameter value.
Comment	Lists the reason for the modification.

4.4.4.3 Sample Window

The Sample Window is used to display detailed measurement information. The information displayed in the Sample Window varies based on the type of analysis performed. To view the Sample Window, open a Sample Table, place the cursor in the row of the Sample Table that contains the desired sample, and select Sample Window from the View menu. The Sample Window is displayed.

TIP: *To view measurement data in real time, place the cursor in the row of the Sample Table that contains the current sample (**********), then open the Sample Window.*

The Sample Window is divided into 3 sections: the Graph, the Parameter Table, and the Injection Table. The sections can be resized by dragging the dividers between the sections.

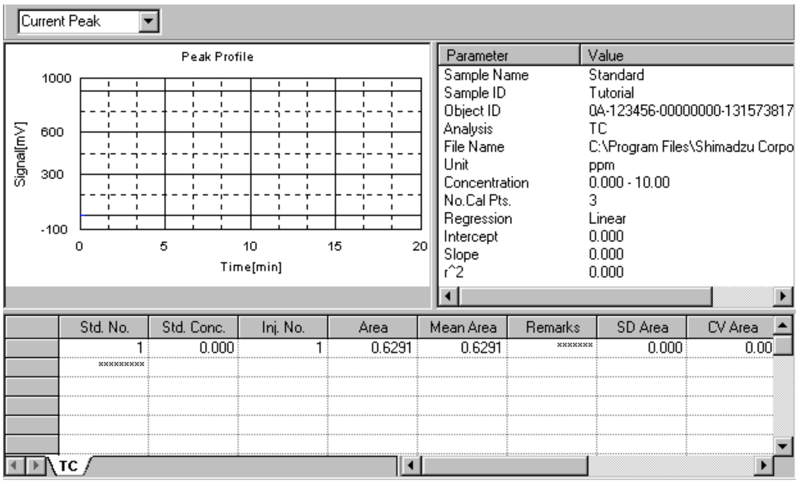


Figure 4.115 Sample Window

Graph

The upper left section of the Sample Window displays the peak profile graph. Change the graph type by choosing from the options on the drop-down list.

Setting Items	Description
Current Peak	Displays the peak profile of the highlighted injection.
All Peaks	Displays all injection peaks for the selected sample.
Cal. Curve	Displays calibration curves (for calibration standards only). This function is not available during real-time analysis

Note: To view the peak profile of the measurement in progress, click the row with the asterisk in the Sample No. column. To view a previous measurement, click the desired row.

Parameter Table

The upper right section of the Sample Window displays sample parameters.

Setting Item	Description
Sample Name	Displays the name of highlighted sample.
Sample ID	Displays the sample identification from the Sample Table.
Object ID	Displays the ID for each set of measured data.
Analysis	Displays the analysis method used for the sample.
Result	Displays the sample concentration.
Dilution Factor	Displays the preparation dilution factor. The result is multiplied by this factor to calculate the final concentration. If the sample was not diluted, do not change the default value of 1.

Injection Table

The lower section of the Sample Window displays the Injection Table, which contains detailed information about the injections performed for the selected sample. Asterisks are displayed in the Injection Table for the injection that is currently being measured. Some columns in the Injection Table use the Merge Cell function to display information that is common to all the injections of a particular sample. If the sample has been analyzed by more than one analysis method, the Injection Table contains several folders displayed as tabs at the bottom of the window. The columns in the Injection Table are described below.

Setting Items	Description
Spl. No.	Displays the sample number.
Inj. No.	Displays the injection number.
Area	Displays the area value.
Mean area	Displays the mean area value for all injections of that sample.
Conc.	Displays the concentration obtained for the injection.
Mean Conc.	Displays the mean concentration obtained for all injections of the sample.
Result	Displays the analysis result.
Notes	Displays any analysis flags or notations associated with that sample.
SD Area	Displays the standard deviation calculated from the area.
CV Area	Displays the coefficient of variation calculated from the area.
SD Conc.	Displays the standard deviation calculated from the concentration.
CV Conc.	Displays the coefficient of variation calculated from the concentration.
Status	Displays the analytical status, either “measuring” or “finished.”
Vial	Displays the vial number (for ASI samples only).
Excluded	Displays the exclusion flag, denoted as “E” for user exclusion of the sample, “ES” for exclusion by the system, or “Not Excluded”.
Modified	Displays the modification flag of “M” if the sample was modified.
Inj. Volume	Displays the injection volume for each injection.
Dilution Factor	Displays the preparation dilution factor used in measurement.
Calibration curve	Displays the calibration curve used.

4.4.4.4 Outlier Test

The Outlier Test (also known as the Grubb test) is used to determine whether a selected injection is an outlier. The test also determines whether the injection with the highest deviation from the mean value is still within an acceptable range. The Outlier Test can only be performed if the sample contains at least three non-excluded injections.

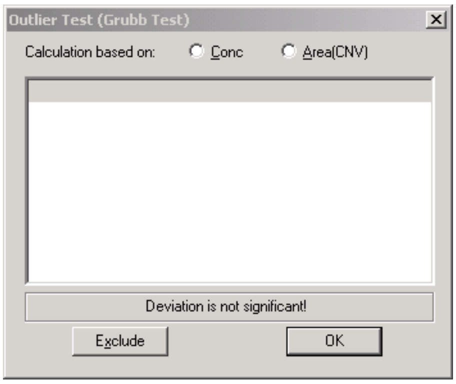


Figure 4.116 Outlier Test (Grubb Test) Window

To perform the Outlier Test, open the Sample Window and select the cells containing the data to be tested. From the View menu, select Outlier Test. The Outlier Test (Grubb Test) window is displayed. Select either Conc. or Area (CNV) for the data source. Exclude the injection that is suspected of being an outlier, then select OK to close the dialog box.

If the sample does not contain enough injections for the test to be performed, an error message is displayed and the Outlier Test (Grubb Test) window will not open.

The Outlier Test (Grubb Test) window contains the parameters listed below.

Setting Items	Description
Result List	Displays the result of the Outlier Test.
Conc. radio button	Select to indicate that test calculations are to be performed based on the concentration data.
Area (CNV) radio button	Select to indicate that test calculations are to be performed based on the area data.
Result	Displays the result of the test.
Exclude button	Click this button to exclude a suspect sample and recalculate the test result.
Suspicious inj.	Indicates the injection suspected of being an outlier, which is identified as the sample with the highest deviation from the mean value.
SD	Displays the standard deviation for all of the injections.
CV	Displays the coefficient of variation for all of the injections.
Grubb Value	Displays the limit value from the Grubb table.
Test Value	Displays the calculated Outlier Test value.

4.4.4.5 Properties

The View>Properties function displays various windows. The display depends on the content of the cell selected in the sample table. If a range of cells is selected, the upper left cell is considered the active cell. The table below shows the possible selections and the corresponding Properties windows.

Table 4.7 Properties Windows

Content of Active Cell	Properties Window
Calibration Curve	Calibration Curve Properties window
Method	Sample/Method Properties window
Any other cell for a sample	Sample/Method Properties window
Any other cell of a control sample	Control Sample Properties window

The Properties window contains options for viewing and modifying the method, calibration curve, and sample properties. Each Properties window consists of several tabs. The Calibration Curve Properties window tabs are shown and described below.

Calibration Curve Properties Window

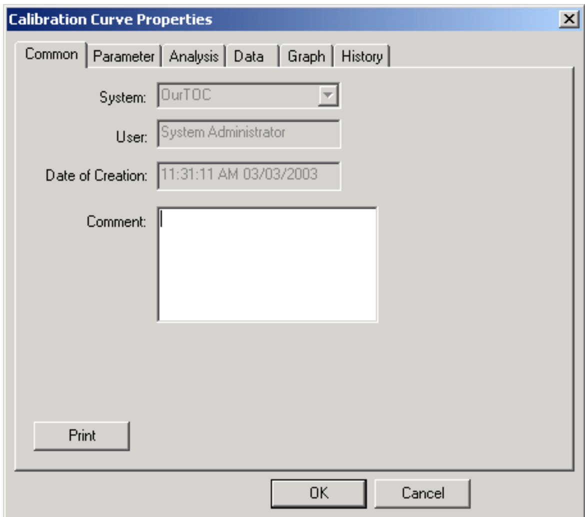


Figure 4.117 Calibration Curve Properties Window: Common Tab

Common Tab

The Common tab of the Calibration Curve properties window contains information about the calibration curve.

Setting Item	Description
System	Displays the system used to create the calibration curve. This field cannot be edited.
User	Displays the name of the user who created the calibration curve.
Date of Creation	Displays the date and time the calibration curve was created.
Comment	A comment regarding the calibration curve may be entered here.
Print	Prints the calibration curve information.

Parameter Tab

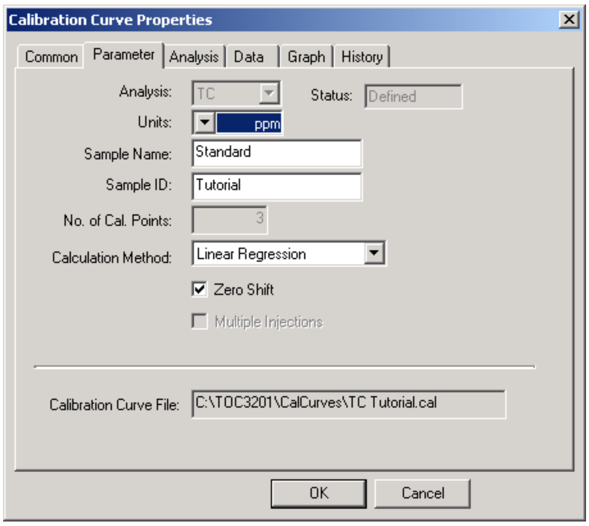


Figure 4.118 Calibration Curve Properties Window: Parameter Tab

Setting Item	Description
Analysis	Displays the analysis method used to create the calibration curve. This field cannot be edited.
Unit	Displays the concentration units. This field can be edited.
Sample Name	Displays the default sample name used for the calibration standards (64 characters maximum). This field can be edited.
Sample ID	Displays the default sample ID (64 characters maximum). This field can be edited.
No. of Cal. Points	Displays the number of calibration standards used. This field cannot be edited.
Calculation Method	Displays the calibration curve calculation method. This field can be edited by selecting a different option from the drop-down list. Two calculation methods are available: point-to-point and linear regression. For both types, a curve representing the relationship between detector response and concentration is displayed. The point-to-point curve fit draws a straight line between adjacent data points and considers each line segment to be a separate calibration line governed by its own equation. A linear regression statistically determines the line that best fits the pattern of all data points. The number used to describe the agreement between the calculated line and the data points is called the Coefficient of Determination (r^2). A coefficient of 1 indicates that the line fits the data points perfectly. The general equation for a straight line is: $Y = mX + b$ where: Y = Y-axis data point X = X-axis data point m = slope of line b = Y-axis intercept point
Zero Shift	Select Zero Shift to shift the regression curve through the origin. This option does not change the slope of the line. Use this option as a correction when the TC content in the Zero water used to prepare the calibration standards is too large to ignore.
Calibration Curve File	Displays the file name of the calibration curve. This field cannot be edited.

Analysis Tab

The Analysis tab displays additional analysis parameters.

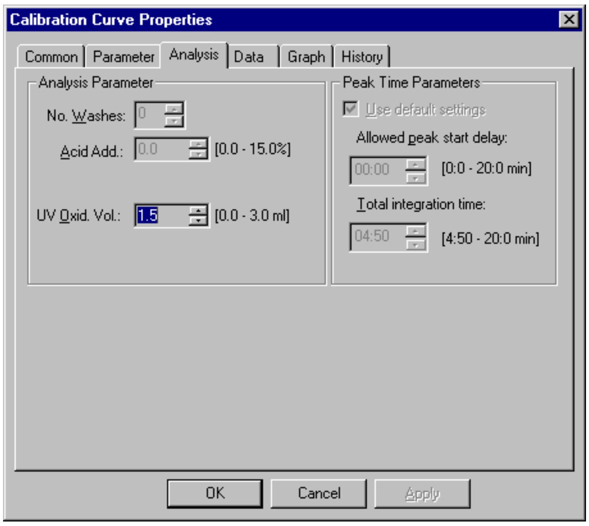


Figure 4.119 Calibration Curve Properties Window: Analysis Tab

Analysis Parameters

Setting Item	Description
No. Washes	Enter the number of times (0-10) the syringe is washed with standard prior to the first injection. If the Auto Dilution option was selected, the standard is diluted and the syringe washed with the diluted standard.
Acid Add	Enter the quantity of acid to be added to each standard prior to injection. This value is expressed as a percent of the sample volume. This option is available only for NPOC or related analyses.
Sparge Time	Enter the pre-sparge time (0-20 minutes). This option is only available when the external sparge kit is enabled and the analysis type is NPOC.
UV Oxid. Vol.	Enter the volume of oxidizer to be added.

Peak Time Parameters

Setting Item	Description
Use default settings	This option is selected if the default settings are used for peak integration.
Allowed peak start delay	Displays the amount of time (in minutes) that can elapse before the analysis is stopped because a peak is not detected.
Total integration time	The total integration time is the amount of time (in minutes) that analysis continues after the injection. If the peak end is determined prior to the set time, integration stops and the next injection is made. Enter a number between 4:50 and 20:00.

Data Tab

The Data tab displays information about each calibration standard used in the calibration curve.

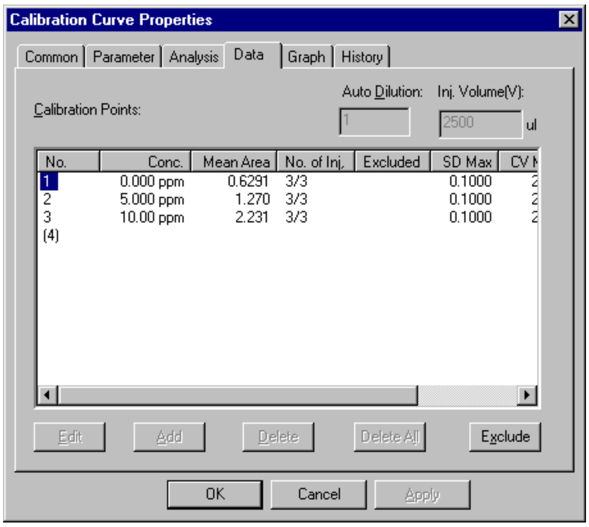


Figure 4.120 Calibration Curve Properties Window: Data Tab

Setting Item	Description
Auto Dilution	Displays the auto dilution factor.
Inj. Volume	Displays the injection volume, which varies depending on the analysis type.

The columns of the Calibration Points window are described below.

Table 4.8 Data Tab Columns

Column Name	Description
No.	Displays the calibration point number.
Conc.	Displays the concentration of the calibration point.
Mean Area	Displays the mean area value counts from all non-excluded injections.
No. of Injections	Displays the minimum and maximum number of injections.
Excluded	"E" indicates that a calibration point has been excluded.
SD Max	Displays the maximum standard deviation limit.
CV Max	Displays the maximum coefficient of variation limit.
SD	Displays the calculated standard deviation.
CV	Displays the calculated coefficient of variation.

Additional buttons are displayed below the data columns, and can be used to manipulate the calibration parameters. The buttons are described below. Refer to Section 4.4.2.2 "Calibration Curve" for more detailed descriptions.

Button	Function
Edit	Edit the parameters for the selected calibration point.
Add	Add a new calibration point.
Delete	Delete the selected calibration point.
Delete All	Delete all calibration points.
Exclude	Exclude the selected calibration point.

Graph Tab

The Graph tab displays the calibration curve in a graphical format.

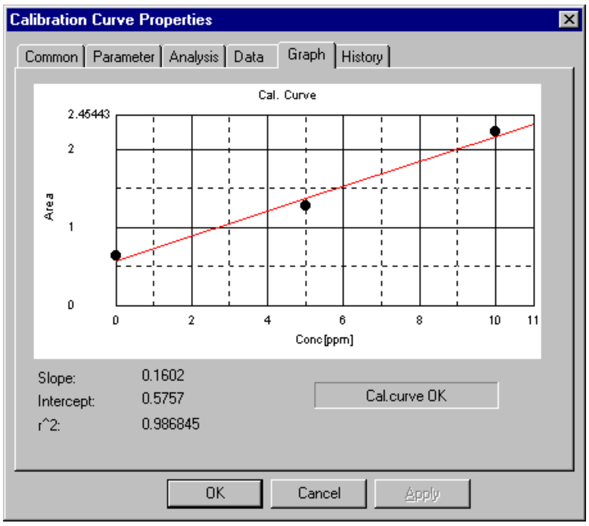


Figure 4.121 Calibration Curve Properties Window: Graph Tab

Setting Item	Description
Slope/Intercept/r^2	The values for Slope, Intercept, and r^2 are displayed for the selected calibration curve.

History Tab

If the History Log was enabled when the calibration curve was created, the History tab displays a list of changes made to the calibration curve parameters. If the History Log was not previously enabled, it can be enabled on the History tab.

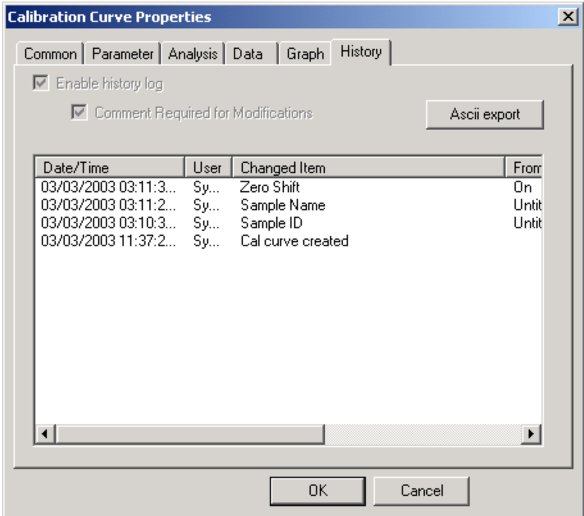


Figure 4.122 Calibration Curve Properties Window: History Tab

Setting Item	Description
Enable history log	Select this option to enable the software to monitor all modifications to the system. If the History Log is not required, it need not be enabled, as the software operates properly without it.
User has to comment all modifications	Select this option to require comments or approval for all modifications. If the History Log is not required, it need not be enabled, as the software operates properly without it.
Ascii Export	The displayed items in the list are exported as a tab-delimited text file.

A list of the Comment dialog box contents is shown below:

Table 4.9 Comment Dialog Box Contents

Item	Description
Date/Time	Displays the date and time of the modification.
User	Displays the user who made the modification.
Changed item	Describes the parameter that changed.
From	Displays the previous parameter value.
To	Displays the new parameter value.
Comment	Lists the reason for the modification.

4.4.4.6 Customize

The View>Customize window contains two tabs, one that displays or hides the toolbars and one that customizes the toolbar content.

Toolbars Tab

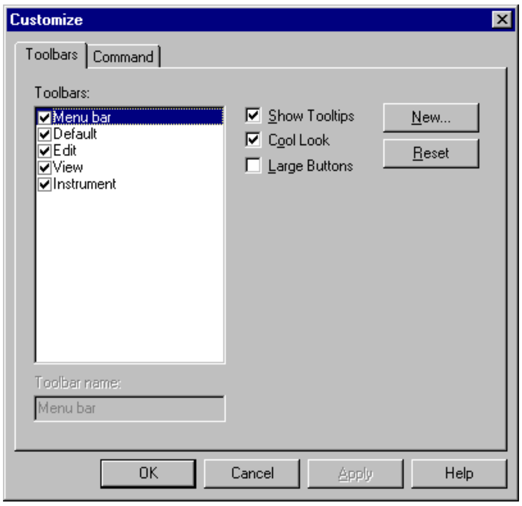


Figure 4.123 Customize Window: Toolbars Tab

Select the desired toolbars from the list. All items are selected by default.

Option	Description
Show Tooltips	Displays tooltips when the mouse cursor is positioned over the toolbar button.
Cool Look	Select this option to display the toolbar buttons as flat buttons. This item is selected by default.
Large Buttons	Select this option to display the toolbar buttons in a larger size.
New button	Click this button to create a user-defined toolbar button.
Reset button	Click this button to reset the toolbar display to its original state or delete a user-defined toolbar button.

Command Tab

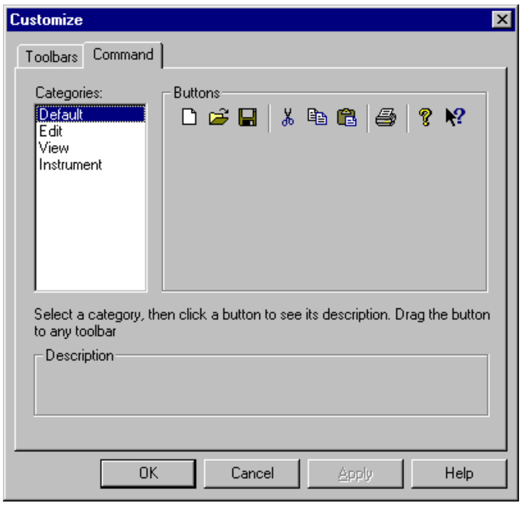


Figure 4.124 Customize Window: Command Tab

Option	Description
Categories	Displays the available toolbars.
Buttons	Displays the buttons in the selected toolbar. The buttons can be moved by dragging up to any toolbar.
Description	Displays a description of the selected button.

4.4.4.7 Toolbar

The View>Toolbar function is used to select or deselect individual toolbars for display. Toolbar display options (such as button size or appearance) can also be set using this command.

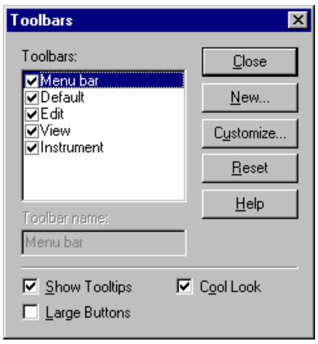


Figure 4.125 Toolbar Window

4.4.4.8 Status Bar

The View>Status Bar function toggles the display state of the status bar between Show and Hide. If the status bar is currently displayed, selecting this option will hide the status bar. If the status bar is not displayed, selecting this option will display the status bar.

4.4.4.9 ASI / 8-Port Sampler Vials

This function displays the ASI or 8-Port Sampler dialog box, and allows the user to enter vial numbers for measurement using the ASI or the 8-Port Sampler. To access this command, either create or open a sample table, and select View > ASI / 8-Port Sampler Vials. The Sparge / Acid Addition dialog box opens automatically when the Start command is made. The OK button saves the changes and closes the dialog box.

Using the Autosampler

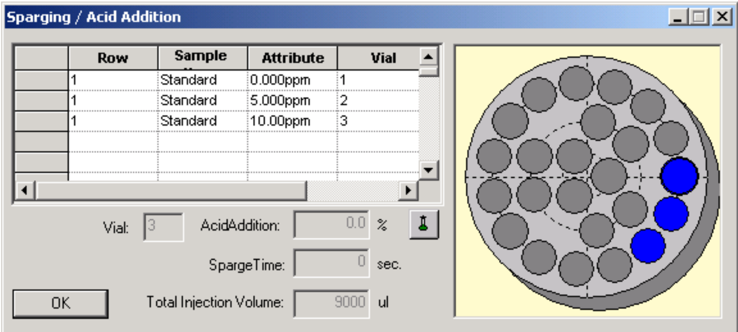


Figure 4.126 Sparge / Acid Addition Dialog Box for the ASI-V

Using One 8-Port Sampler

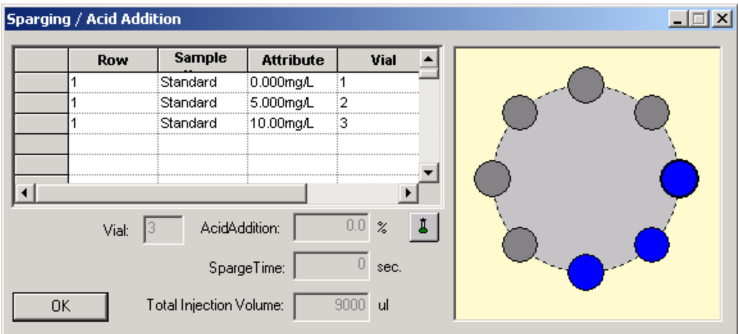


Figure 4.127 Sparge / Acid Addition Dialog Box for One 8-Port Sampler

Using Two 8-Port Samplers

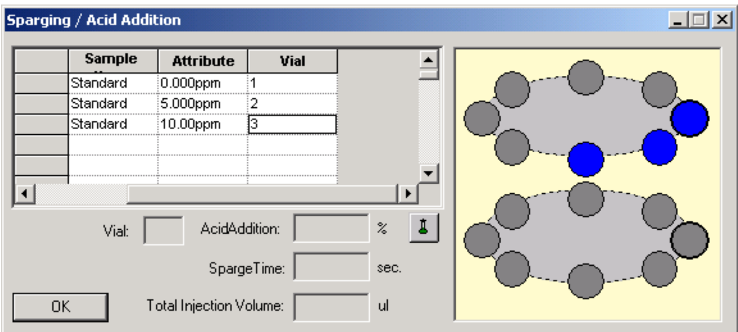


Figure 4.128 Sparge / Acid Addition Dialog Box for Two 8-Port Samplers

When the mouse is positioned over a vial in the drawing of the of ASI or 8-Port Sampler, a tool-tip showing the vial number displayed. Click the vial to display the following items.

Parameter	Description
Vial No.	Displays the currently selected vial number.
Acid Addition rate	Displays the acid addition percentage for each vial.
Sparge time	Displays the sparge time for each vial.
Total injection volume	Displays the total injection volume for each vial.

TIP: *The desired vial position for a sample can be entered either directly into the Vial column or by placing the cursor in the appropriate row of the Sparging/Acid Addition window and then double-clicking the desired position in the tray diagram.*

4.4.4.10 Data Profile

Displays the raw measurement data. The data profile is never modified or processed following the initial collection. It includes the corresponding measurement parameters. The files which were created and named using the object ID, each occupy a row in the sample table, and are stored within the RawData folder in folders named using the date of creation.

The View>Data Profile function is used to view the data profile for a sample. Highlight the desired row of the Sample Table, then select the command.

4.4.5 Insert Menu

The Insert menu contains options for inserting sample groups, calibration curves, individual samples, and control samples into the Sample Table.

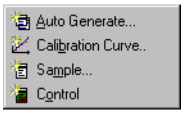


Figure 4.129 Insert Menu

4.4.5.1 Auto Generate

The Auto Generate command opens the Sample Group Wizard, which is used to create a group of unknown samples, control samples, and calibration standards, and then insert the group into the active position of the Sample Table.

Sample Group Wizard (Page 1) Sample Source

The Sample Source window is used to specify the source of sample measurement parameters.

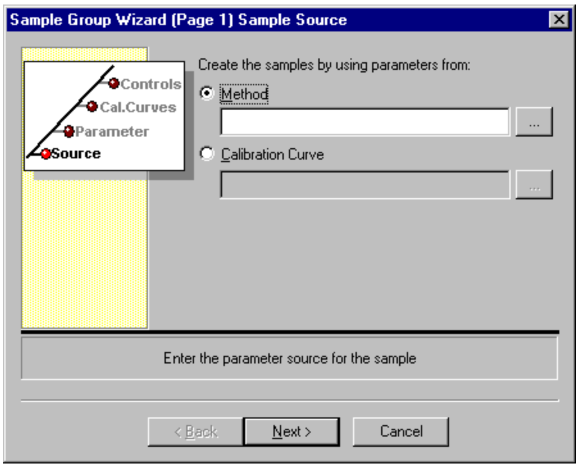


Figure 4.130 Sample Group Wizard (Page 1) Sample Source

Setting Item	Description
Method radio button	Click this option to analyze the samples using the measurement parameters from a specified method. This is the default option.
Method text field	Enter the desired method or select the method using the Browse button.
Calibration Curve radio button	Click this option to analyze the samples using the measurement parameters from a specified calibration curve.
Calibration Curve text field	Enter the desired calibration curve or select the curve using the Browse button.

Sample Group Wizard (Page 2) Sample Parameter

The Sample Parameter window is used to enter basic information about the sample group.

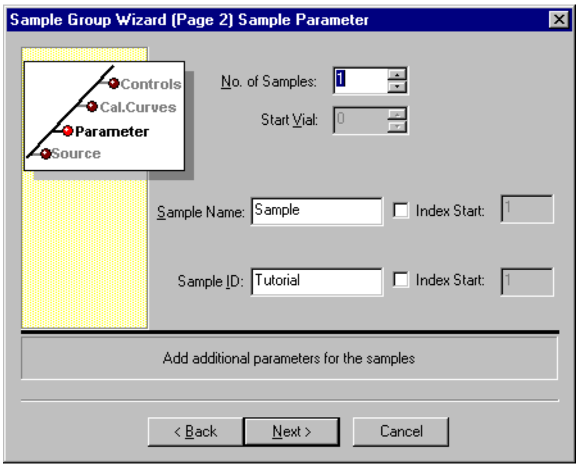


Figure 4.131 Sample Group Wizard (Page 2) Sample Parameter

Setting Item	Description
No. of Samples	Enter the number of samples in the group. Enter an integer ranging from 1 to 200. The default value is 1.
Start Vial	Enter the starting vial position for the sample group. Enter an integer ranging from 0 to 9 or 1 to 100, depending on the sample tray size. The default value is 1. This field is disabled if an autosampler is not a component of the configured system.
Sample Name/Sample ID	The default designations for these fields are obtained from the method or calibration curve identified in Page 1 of the Sample Group Wizard. Enter other names if desired.
Index Start	Select this option to start the counting index. Each sample added receives an increased counting index number.

Sample Group Wizard (Page 3) Calibration Curves

The Calibration Curves window is used to identify the calibration standards in the sample group and to specify their placement in the group. The window is also used to revise the list of calibration curves that are used to quantitate samples in the group.

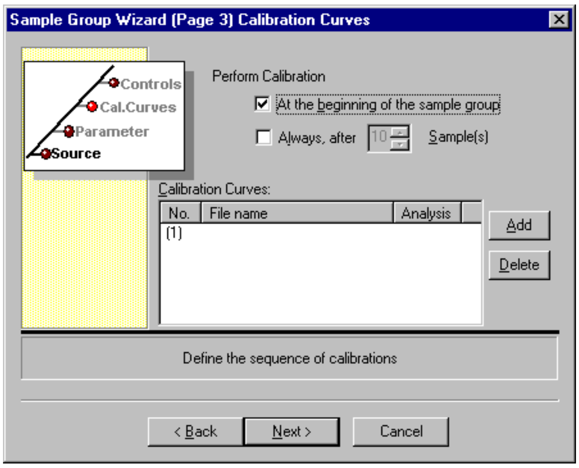


Figure 4.132 Sample Group Wizard (Page 3) Calibration Curves

Setting Item	Description
Perform Calibration	Select one of the available options for when calibration should be performed: *At the beginning of the sample group* or *Always, after (a selected number of) Samples* have been analyzed.
Add button	Use this button to add a calibration curve.
Delete button	Highlight a calibration curve listed in the window and click the Delete button to remove the curve from the sample group.
Calibration Curves	*No.:* Displays the calibration point number. *File name:* Displays the calibration curve name. *Analysis:* Displays the analysis type.

Sample Group Wizard (Page 4) Controls

The Controls window is used to identify the control samples in a sample group and to specify their placement in the sample group.

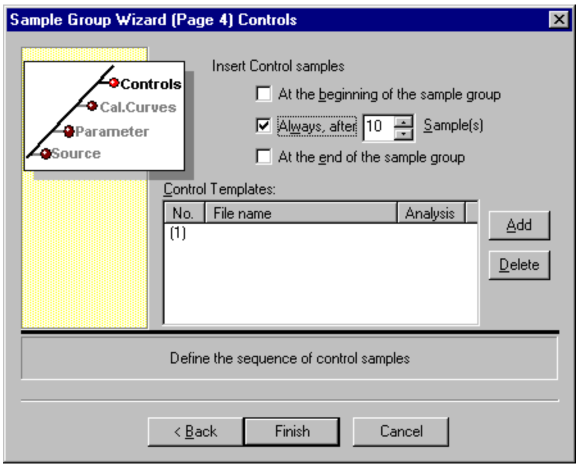


Figure 4.133 Sample Group Wizard (Page 4) Controls

Insert Control Samples

Use the options listed below to insert individual control samples in the sample group.

Setting Items	Description
At the beginning of the sample group	Select this option to add control samples at the beginning of the sample group.
Always, after (number of) Sample(s)	Select this option to add control samples at defined intervals between unknown samples.
At the end of the sample group	Select this option to add control samples to the end of the sample group.

Control Templates

The Control Templates table displays a list of the control sample templates currently assigned to the sample group. The templates specify measurement parameters, evaluation criteria, and failure actions for the control samples. This table is empty by default. Use the options listed below to revise the list of control sample templates associated with the sample group.

Option	Description
Add	Select this option to add a control template to the sample group.
Delete	Select this option to delete a highlighted control template from the list.

After all four pages of the Sample Group Wizard are complete, click the Finish button to save changes and add the sample group to the Sample Table. If invalid parameter values were entered, an error message is displayed and the sample group is not inserted into the Sample Table until the error is corrected.

4.4.5.2 Sample

The Sample command opens the Sample Wizard, which is used to insert sample measurement parameters into an existing Sample Table. The Sample Wizard is not accessible during an analysis.

Sample Wizard (Page 1) Parameter Source

The Parameter Source window is used to specify the source of the sample measurement parameters. The source can either be a method file or a calibration file. When one of these sources is selected, the sample is analyzed using the same measurement parameters as those in the specified file. Some of the source parameters can be edited in subsequent pages of the Sample Wizard. A sample can also be added to the Sample Table without specifying a source, an option that is desirable when a sample is analyzed using different parameters than those specified in the method or calibration files. In this case, unique measurement parameters for the sample are entered on subsequent pages of the Sample Wizard

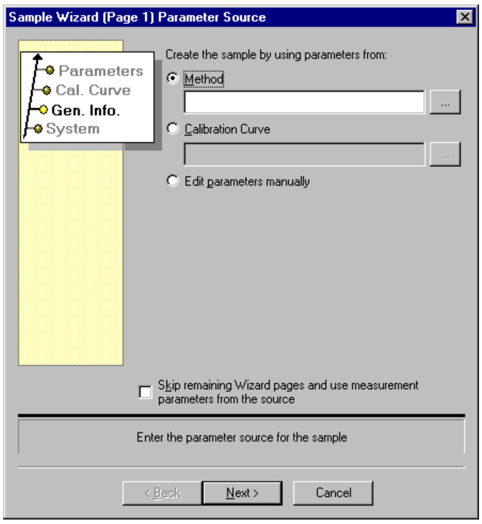


Figure 4.134 Sample Wizard (Page 1) Parameter Source

Parameter	Description
Method radio button	Select this option to use the sample measurement parameters from a specified method. This is the default option. Enter the method name in the text field or use the Browse button to select the file using the File>Open dialog box.
Calibration Curve radio button	Select this option to use the measurement parameters from a specified calibration curve. Enter the calibration curve name in the text field, or use the Browse button to select the file using the File>Open dialog box.
Edit parameters manually radio button	Select this option to insert a sample without defining a source. Sample measurement parameters are entered in pages 1-12 of the Sample Wizard.
Skip remaining Wizard pages and use measurement parameters from the source	Select this option to accept all measurement parameters from the source file. The Next button becomes a Finish button, and subsequent pages of the Sample Wizard are not displayed. This option is disabled if *Edit parameters manually* is selected.

Sample Wizard (Page 2) Analysis Information

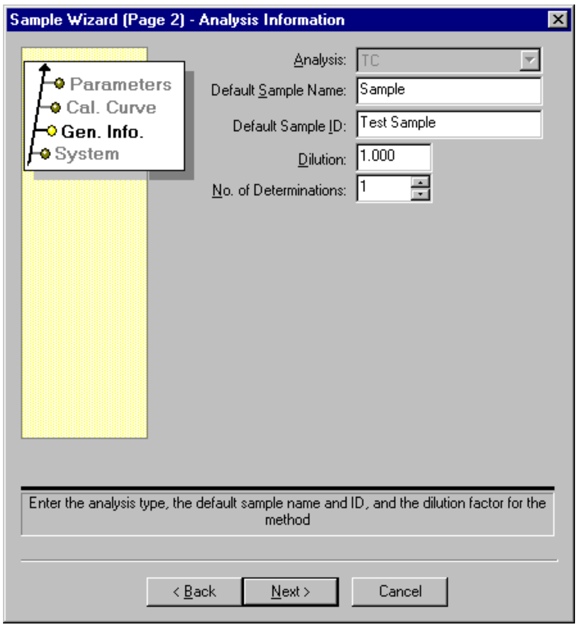


Figure 4.135 Sample Wizard (Page 2) Analysis Information

Parameter	Description
Analysis	Select the analysis type from the drop-down list.
Default Sample Name	Enter the sample name.
Default Sample ID	Enter the sample identification.
Dilution	Enter the preparation dilution factor.
No. of Determinations	Use the spin controls to enter the number of times the sample is measured. Determinations and injections are different; each determination is made up of one or more injections. Enter an integer from 1 to 10.

Note: For combined analyses, some Sample Wizard windows may appear more than once so that parameter modifications specific to each analysis type can be entered. The analysis type that is associated with each Sample Wizard window is found in the Analysis box at the top of the window.

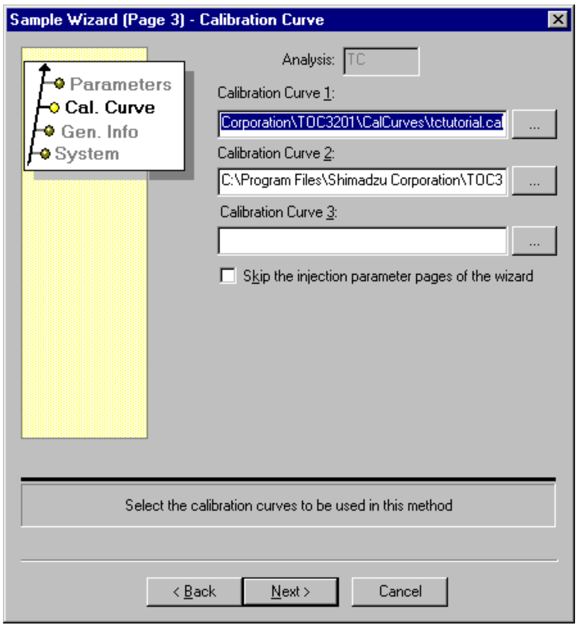


Figure 4.136 Sample Wizard (Page 3) Calibration Curve

Sample Wizard (Page 3) Calibration Curve

The Calibration Curve window is used to identify the calibration curve(s) used to calculate the sample concentration. If more than one curve is identified, the software automatically selects the curve to be used in calculating sample concentration. Refer to Section 6.2.4 "Automatic Selection of the Optimal Calibration Curve" for details.

Parameter	Description
Analysis	Displays the analysis type. This field cannot be edited.
Calibration Curve 1/2/3	Enter the name of the calibration curve or click the Browse button to select a file using the File>Open dialog box.
Skip the injection parameter pages of the wizard	Select this item to skip the Measurement Parameters window of the Sample Wizard. Use this option when the sample measurement parameters are the same as those used for calibration curve measurements.

Sample Wizard (Page 4) Injection Parameters

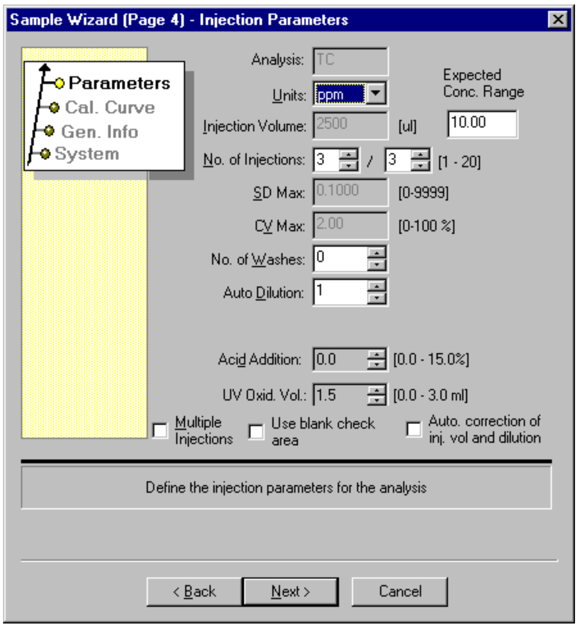


Figure 4.137 Sample Wizard (Page 4) Injection Parameters

Parameter	Description
Analysis	Displays the analysis type. This field cannot be edited.
Units	Enter the concentration units.
Injection Volume	Enter the injection volume.
Expected Conc. Range	Enter the maximum expected sample concentration.
No. of Injections	Enter the minimum and maximum number of injections for the sample.
SD Max	Enter the maximum acceptable standard deviation. If this value is met, no additional injections are required. If both this value and the CV Max value are exceeded, the sample is automatically reinjected up to the maximum number of times. The SD Max field is disabled if the No. of Injections is 1 or if the minimum is equal to the maximum.
CV Max	Enter the maximum acceptable coefficient of variation. If this value is met, no additional injections are required. If both this value and the SD Max value are exceeded, the sample is automatically reinjected up to the maximum number of times. The CV Max field is disabled if the No. of Injections is 1 or if the minimum is equal to the maximum.
No. of Washes	Enter the number of times the syringe is washed with sample prior to the first injection. If the Auto Dilution option is selected, the sample is diluted and the syringe washed with the diluted sample.
Auto Dilution	Enter a dilution factor for the sample. The instrument automatically dilutes the sample by this factor, and the result is multiplied by the factor to obtain the final concentration. The default Auto Dilution value is 1.
Sparge Time	Enter the sparge time.

Parameter	Description
Acid Addition	Enter the desired quantity of acid to be added. This value is expressed as a percent of the sample volume. This option is available only for NPOC or related analyses.
UV Oxid. Vol.	Sets the volume of wet chemical oxidizer that will be added.
Use blank check area	Select this option to subtract the blank check area from the measured area of the sample. This option is only available for TC and NPOC analyses.
Auto. correction of inj. vol. and dilution	Select this option to enable the instrument to automatically adjust the injection volume and dilution factor when the peak height for the initial injection exceeds full scale. After the adjustment, measurement is repeated.

Sample Wizard (Page 5) Peak Time Parameters

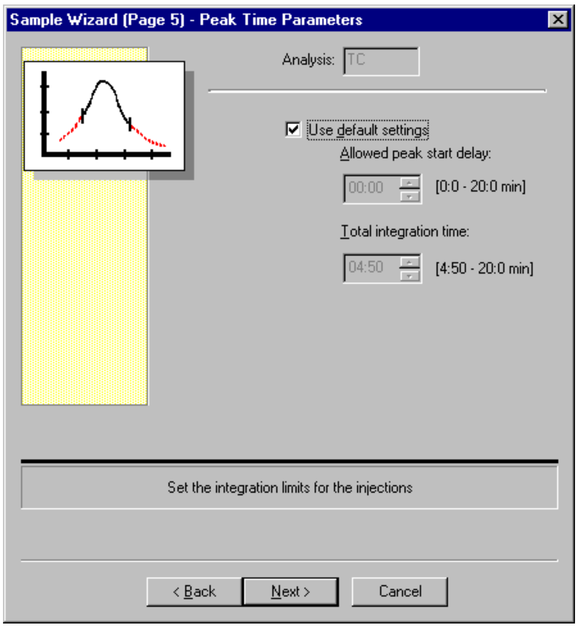


Figure 4.138 Sample Wizard (Page 5) Peak Time Parameters

Parameter	Description
Analysis	Displays the analysis type. This field cannot be edited.
Use default settings	Select this option to use the default integration parameters. When this option is selected, all other options in this window are disabled.
Allowed peak start delay	The allowed peak start delay is the amount of time (in minutes) that can elapse before the analysis is stopped because a peak is not detected. Enter a number between 0 and 20:00.
Total integration time	The total integration time is the amount of time (in minutes) that analysis continues after the injection. If the peak end is determined prior to the set time, integration stops and the next injection is made. Enter a number between 4:50 and 20:00.

Sample Wizard (Page 6) Calibration Curve

A second Calibration Curve window is only displayed when more than one analysis type has been specified. This window is the same as Page 3 of the Sample Wizard, which is described above. This screen is displayed when the measurement type is TOC. Verify the analysis type in the Analysis text box at the top of the page and make the appropriate selections.

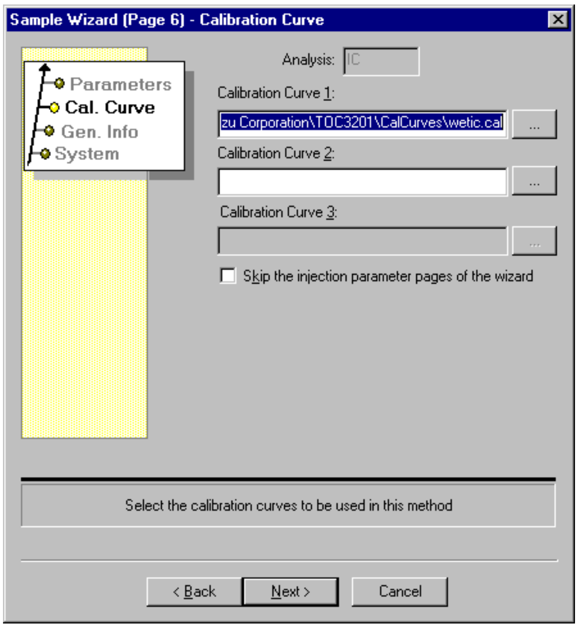


Figure 4.139 Sample Wizard (Page 6) Calibration Curve Window (Second Analysis)

Sample Wizard (Page 7) Injection Parameters

A second Injection Parameters window is only displayed when more than one analysis type has been specified. This window is the same as Page 4 of the Sample Wizard, which is described above. This screen is displayed when the measurement type is TOC. Verify the analysis type in the Analysis text box at the top of the page and make the appropriate selections.

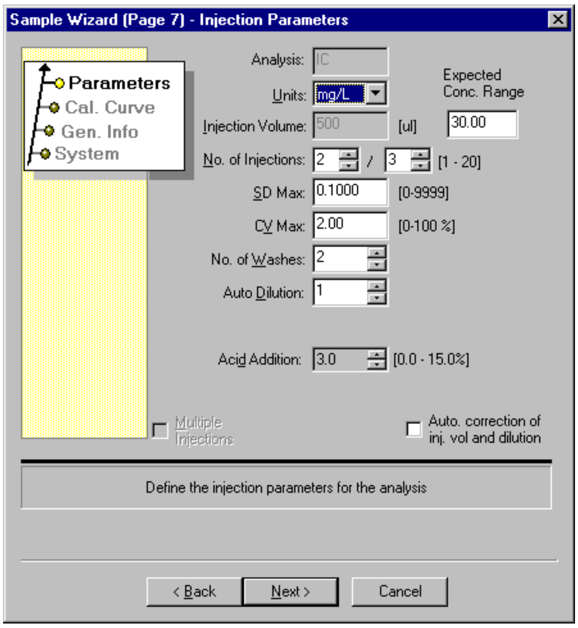


Figure 4.140 Sample Wizard (Page 7) Injection Parameters Window (Second Analysis)

Sample Wizard (Page 8) Peak Time Parameters

A second Peak Time Parameters window is only displayed when more than one analysis type has been specified. This window is the same as Page 5 of the Sample Wizard, which is described above. This screen is displayed when the measurement type is TOC. Verify the analysis type in the Analysis text box at the top of the page and make the appropriate selections.

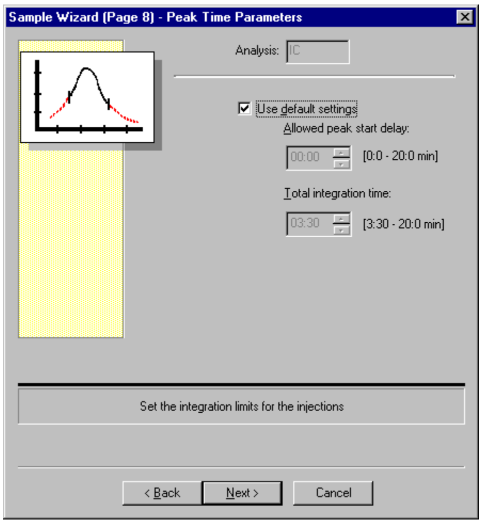


Figure 4.141 Sample Wizard (Page 8) Peak Time Parameters Window (Second Analysis)

Sample Wizard (Page 9) USP/EP

The USP/EP window is displayed only for TC or NPOC methods, and is used to enable samples to be checked in accordance with USP/EP standards. Select the check box to enable the function.

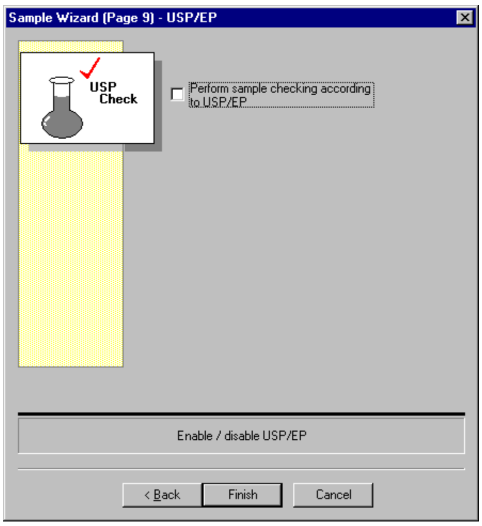


Figure 4.142 Sample Wizard (Page 9) USP/EP

4.4.5.3 Control

The Control command is used to insert a control sample into an existing Sample Table.

Note: A control sample template must be created before a control sample can be inserted into a Sample Table. A control sample template has a file extension of *.tpl and specifies measurement parameters for the Control Sample.

The following subsections describe the procedures for creating a control sample template and for inserting a control sample into a Sample Table.

Creating a Control Sample Template

Control sample templates are created using the Control Sample Wizard. To access the Control Sample Wizard select File>New>Control Sample Template. The Control Sample Wizard consists of several windows, each of which defines specific parameters for the control sample template.

Control Sample Wizard (Page 1) System Information

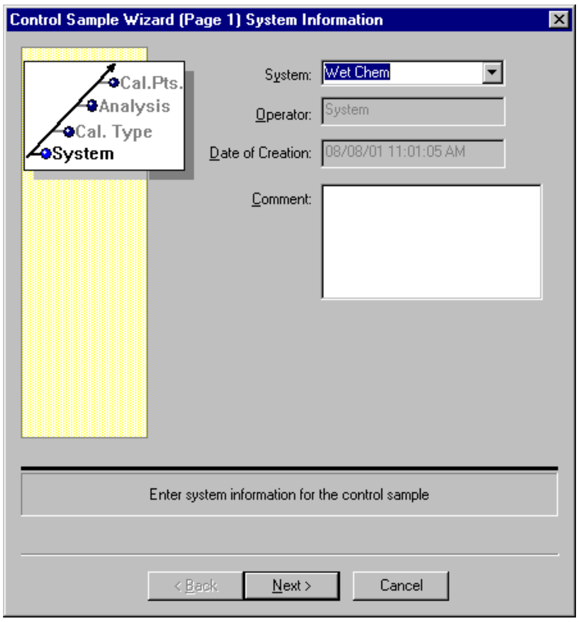


Figure 4.143 Control Sample Wizard (Page 1) System Information

Parameter	Description
System	Select the instrument.
Operator	Displays the current operator name. This field cannot be edited.
Date of Creation	Displays the current date and time. This field cannot be edited.
Comment	Enter a comment (512 characters maximum).

Control Sample Wizard (Page 2) Control Sample Type

The Control Sample Type window is used to identify the type of control sample.

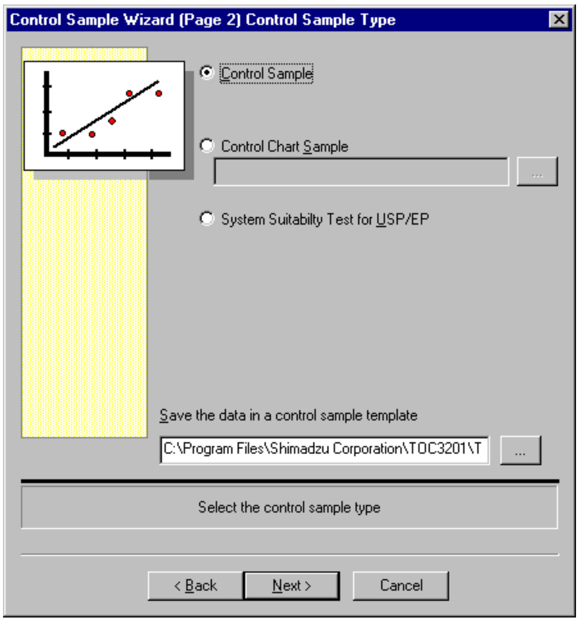


Figure 4.144 Control Sample Wizard (Page 2) Control Sample Type

Parameter	Description
Control Sample	Select this option to define individual sample parameters. This is the default option.
Control Chart Sample	Select this option to add the control sample to a defined control chart. Enter the file name or use the Browse button to select the control chart file from the File>Open dialog box.
System Suitability Test for USP/EP	Select this option to check the control sample using USP/EP criteria.
Save the data in a control sample template	Enter a file name or use the Browse button to select the control sample template from the File>Open dialog box.

Control Sample Wizard (Page 3) Parameter Source

The Parameter Source window is used to specify the source of the control sample measurement parameters. The source can either be a method file or a calibration file. When one of these sources is selected, the control sample is analyzed using the same measurement parameters as those in the specified method or calibration file. Some of the source parameters can be edited in subsequent pages of the Control Sample Wizard. A control sample can also be added to the Sample Table without specifying a source, an option that is desirable when a control sample is analyzed using different parameters than those specified in the method or calibration files. In this case, unique measurement parameters for the sample are entered on subsequent pages of the Control Sample Wizard.

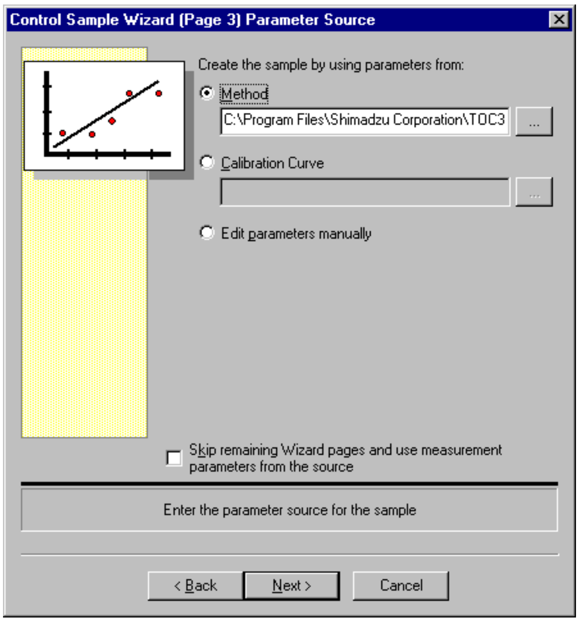


Figure 4.145 Control Sample Wizard (Page 3) Parameter Source

Parameter	Description
Method radio button	Select this option to use the measurement parameters from a specified method. Enter the method name in the text field, or use the Browse button to select the file using the File>Open dialog box. This is the default option.
Calibration Curve radio button	Select this option to use the measurement parameters from a specified calibration curve. Enter the calibration curve name in the text field, or use the Browse button to select the file using the File>Open dialog box.
Edit parameters manually radio button	Select this option to insert a control sample without defining a source. Control sample measurement parameters are entered in subsequent pages of the Control Sample Wizard.
Skip remaining Wizard pages and use measurement parameters from the source	Select this option to accept all measurement parameters from the source file. The Next button becomes a Finish button, and subsequent pages of the Control Sample Wizard are not displayed. This option is disabled if *Edit parameters manually* is selected.

Control Sample Wizard (Page 4) Analysis Parameters

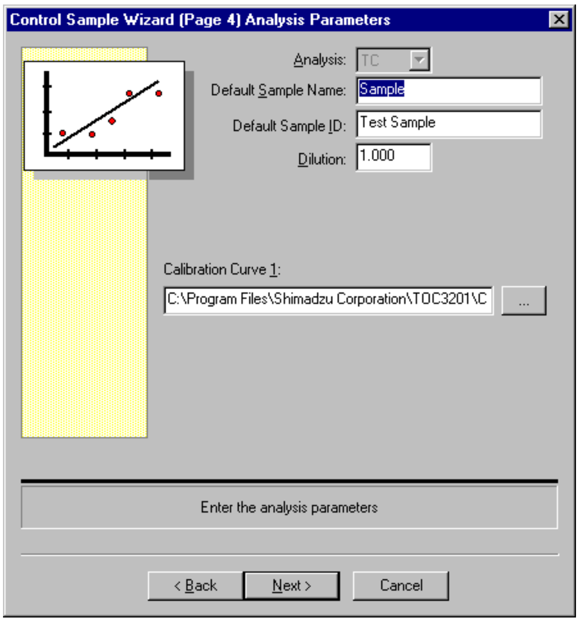


Figure 4.146 Control Sample Wizard (Page 4) Analysis Parameters

Parameter	Description
Analysis	Select the analysis type from the drop-down list. If a source method or calibration file was specified on Page 3 of the Control Sample Wizard, this field is disabled and the applicable analysis type is displayed.
Default Sample Name	Enter the sample name. If a source method or calibration file was specified on Page 3 of the Control Sample Wizard, the default sample name from the source is displayed.
Default Sample ID	Enter the sample identification. If a source method or calibration file was specified on Page 3 of the Control Sample Wizard, the default sample identification from the source is displayed.
Dilution	Enter the dilution factor. If a source method or calibration file was specified on Page 3 of the Control Sample Wizard, the default dilution factor is displayed.
Calibration Curve	Enter the calibration file name that is used to calculate control sample concentration. If a source method or calibration file was specified on Page 3 of the Control Sample Wizard, the default calibration curve name from the source is displayed.

Control Sample Wizard (Page 5) Injection Parameters

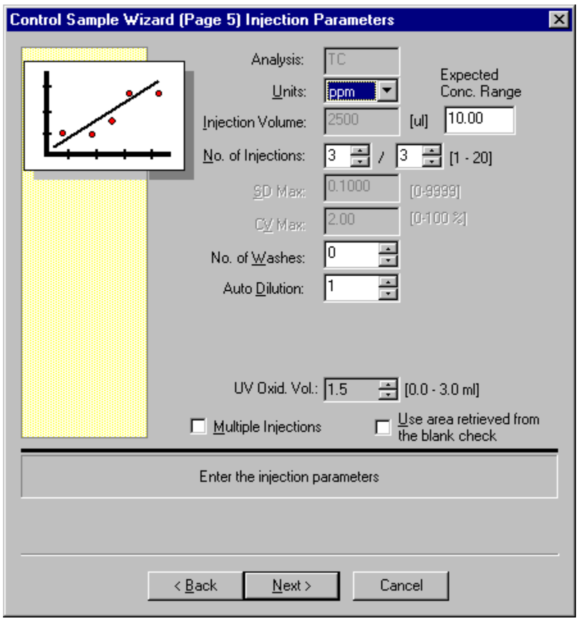


Figure 4.147 Control Sample Wizard (Page 5) Injection Parameters

Parameter	Description
Analysis	Displays the analysis type. This field cannot be edited.
Units	Select the concentration units.
Injection Volume	Enter the injection volume.
Expected Conc. Range	Enter the maximum expected concentration.
No. of Injections	Enter the minimum/maximum number of injections for the control sample. The setting range is from 1-20.
SD Max	Enter the maximum acceptable standard deviation. If this value is met, no additional injections are required. If both this value and the CV Max value are exceeded, the control sample is automatically reinjected up to the maximum number of times. The SD Max field is disabled if the No. of Injections is 1 or if the minimum is equal to the maximum.
CV Max	Enter the maximum acceptable coefficient of variation. If this value is met, no additional injections are required. If both this value and the SD Max value are exceeded, the control sample is automatically reinjected up to the maximum number of times. The CV Max field is disabled if the No. of Injections is 1 or if the minimum is equal to the maximum.
No. of Washes	Enter the number of times the syringe is washed with sample prior to the first injection. If the Auto Dilution option is selected, the sample is diluted and the syringe washed with the diluted sample.

Parameter	Description
Auto Dilution	Enter a dilution factor for the samples. The instrument automatically dilutes the control sample by this factor, and the result is multiplied by the factor to obtain the final concentration. The default Auto Dilution value is 1.
Acid Addition	Enter the desired quantity of acid to be added. This option is available only for NPOC and related analyses.
Sparge Time	Enter the sparge time.
UV Oxid. Vol.	Enter the volume of the wet chemical oxidizer to be added.
Use area retrieved from the blank check	Select this option to subtract the blank check area from the measured area of the control sample.

Control Sample Wizard (Page 6) Peak Time Parameters

The Peak Time Parameters window is used to set peak detection parameters for the control samples.

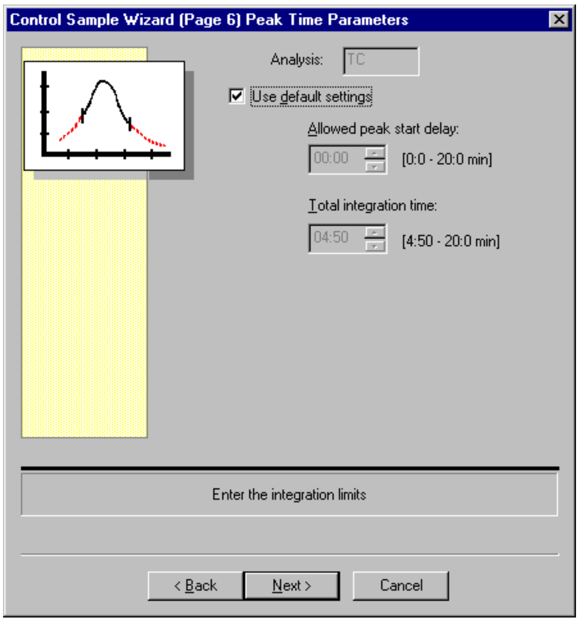


Figure 4.148 Control Sample Wizard (Page 6) Peak Time Parameters

Setting Item	Description
Use default settings	Select this option to use the default integration parameters. When this option is selected, all other options in this window are disabled.
Allowed peak start delay	The allowed peak start delay is the amount of time (in minutes) that can elapse before the analysis is stopped because a peak is not detected. Enter a number between 0 and 20:00.
Total integration time	The total integration time is the amount of time (in minutes) that analysis continues after the injection. If the peak end is determined prior to the set time, integration stops and the next injection is made. Enter a number between 4:50 and 20:00.

Control Sample Wizard (Page 7) History

The History window is used to enable the History Log. The History Log records all modifications to the control sample template properties. The History Log can be set to require users to enter comments for each modification.

Note: Once the History Log is enabled, it cannot be disabled. All modifications to the control sample template are tracked. If the mandatory comment option is selected, the user is required to enter a comment each time any parameter in the control sample template is modified.

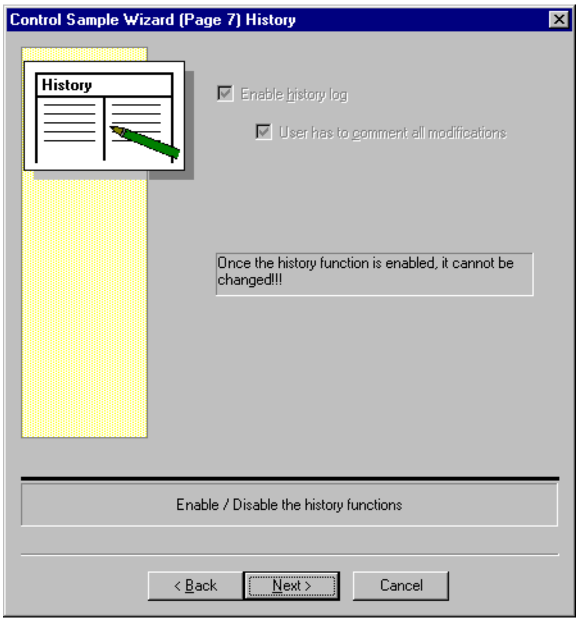


Figure 4.149 Control Sample Wizard (Page 7) History

Setting Item	Description
Enable history log	Select this option to enable the software to monitor all modifications to the control sample template. Once this is enabled, it cannot be disabled. If the History Log is not required, it need not be enabled, as the software operates properly without it.
User has to comment all modifications	Select this option to require comments or approval for all modifications.

Control Sample Wizard (Page 8) Control Checking

The Control Checking window is used to select options for control checks and control limits. This window is not displayed if the USP/EP system suitability criteria are used because the control limits are already defined.

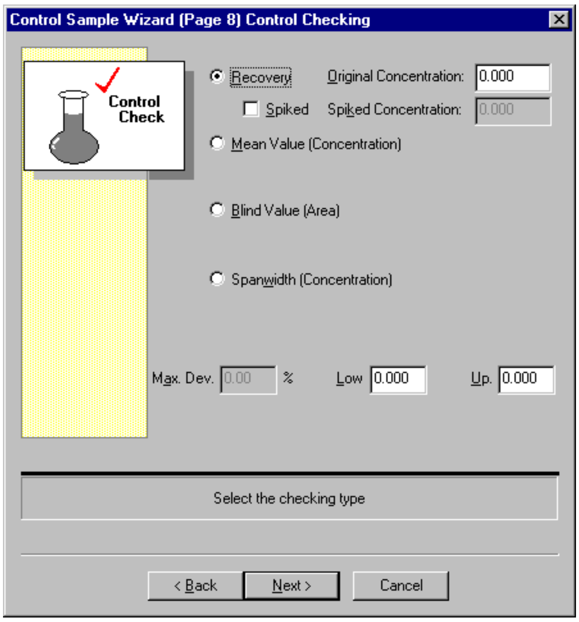


Figure 4.150 Control Sample Wizard (Page 8) Control Checking

Setting Option	Description
Recovery	Select this option to check recovery amounts in control samples.
Spiked	Select this option to use an original and a spiked sample to calculate the recovery amount. This option is enabled only when the Recovery option is selected.
Original Concentration	Enter the original concentration of the spiked sample. This field is enabled only when the Recovery option is selected.
Spiked Concentration	Enter the concentration of the spiked sample. This field is enabled only when the Recovery and Spiked options are selected.
Mean Value (Concentration)	Select this option to check the mean value of the measured concentrations.
Blind Value (Area)	Select this option to check the mean value of the measured area values.
Spanwidth (Concentration)	Select this option to check the deviation of the measured concentrations.
Max. Dev.	Enter the maximum acceptable deviation. This field is enabled only when the Spanwidth option is selected.
Low	Enter the lower limit of the valid control range if the Blind Value option is selected.
Up	Enter the upper limit of the valid control range if the Blind Value option is selected.

Control Sample Wizard (Page 9) Failure Action

The Failure Action window is used to specify the actions taken when a control sample exceeds the specified control limits.

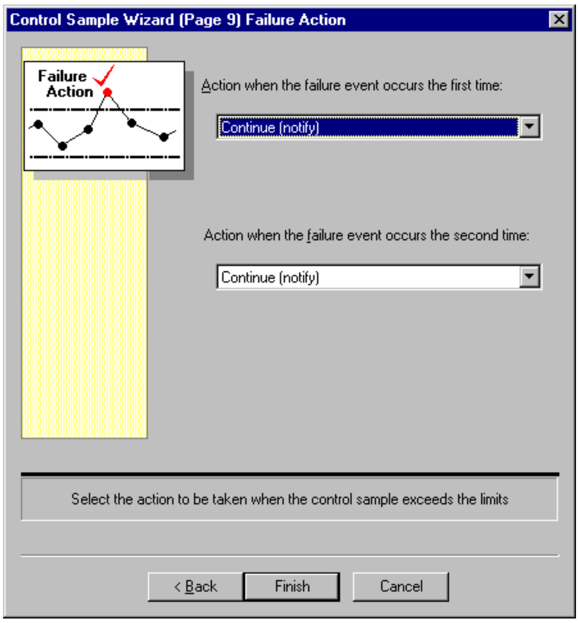


Figure 4.151 Control Sample Wizard (Page 9) Failure Action

Setting Item	Description
Action when the failure event occurs the first time	Select the action to be taken the first time a control sample exceeds the control limits. Options available from the drop-down list are: *Continue (notify):* Select this option to allow the analysis sequence to continue normally and display a failure message in the Notification bar. *Stop (whole sample run):* Select this option to stop the sample run and display a failure message in the Notification bar. *Repeat (from last control or calibration):* Select this option to automatically reanalyze all samples analyzed after the previous valid control sample or calibration curve (whichever occurred most recently) and display a failure message in the Notification bar.
Action when the failure event occurs the second time	Select the action to be taken the second time a control sample exceeds the control limits. The options available from the drop-down list are *Continue* and *Stop*. The *Repeat* option is not available.

Note: Save the control sample template and close the Control Sample Wizard by clicking the Finish button.

Inserting a Control Sample into the Sample Table

Once the control sample template has been created, a control sample can be entered into the Sample Table by following the procedure below.

Procedure

1. Open the Sample Table and place the cursor in the row where the control sample will be inserted.

2. From the Insert menu, select Control.

   The Open dialog box displays the available control sample template files.

3. Select the appropriate control sample template and click the Open button.

   The Open dialog box closes and the Sparging/Acid Addition window is displayed.

4. Enter the ASI vial position for the control sample into the appropriate cell in the Sparging/Acid Addition window.

5. Click the OK button.

   The Sparging/Acid Addition window closes.

6. Scroll through the Sample Table to verify that the control sample has been inserted at the correct location.

4.4.6 Instrument Menu

The Instrument menu contains commands for connecting, starting, and stopping the instrument, and for instrument maintenance functions.

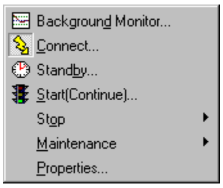


Figure 4.152 Instrument Menu

4.4.6.1 Background Monitor

The Background Monitor command is used to open the Background Monitor window, which displays the status of the autosampler and all installed instrument detectors. The real time signal and ready status for various detectors can be viewed by toggling between the individual instrument tabs. The command is available only when an instrument is connected to the software. Instrument and communication errors are displayed in the Notification bar. The Background Monitor window cannot be opened during measurement.

TOC Tab

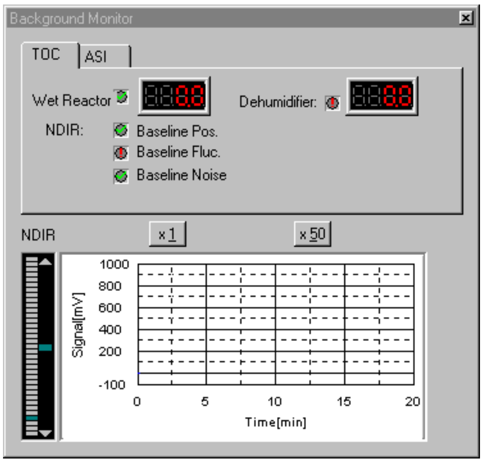


Figure 4.153 Background Monitor Window: TOC Tab

Parameter	Description
Wet Reactor	Displays the temperature of the TC reactor.
Dehumidifier	Displays the temperature of the dehumidifier.
NDIR	*Baseline Pos.:* Displays the status of the NDIR detector baseline position. *Baseline Fluc.:* Displays the status of the NDIR detector baseline fluctuation. *Baseline Noise:* Displays the status of the NDIR detector baseline noise.

The baseline is displayed at the bottom of the TOC tab window.

ASI Tab

The ASI tab is available only if the ASI is connected to the instrument. The ASI Power indicator displays the power status of the ASI. The ASI Cover indicator displays the status of the ASI cover.

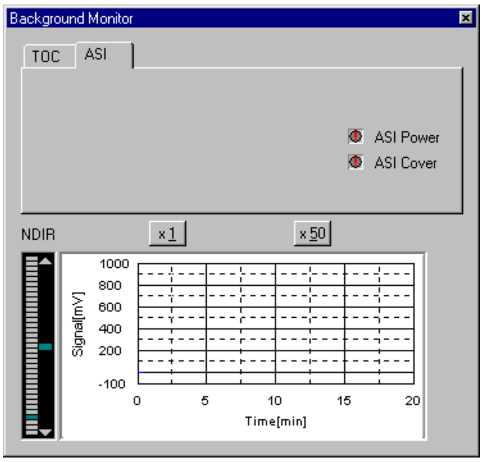


Figure 4.154 Background Monitor Window: ASI Tab

4.4.6.2 Connect

The Connect command is used to connect or disconnect the instrument and the software. If the instrument is not connected, selecting the Connect command opens the communication port, initializes the instrument, and downloads and checks the instrument parameters. During this process, the Sequence window displays the progress of the connection. To abort the Connect operation while it is in progress, click the Cancel button.

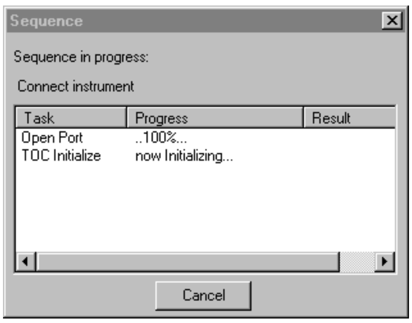


Figure 4.155 Sequence Window

The bottom portion of the Sequence window displays a list of tasks that are performed during the Connect process. The progress and result of each task are shown in columns to the right of the task list. The Sequence window closes when the connection process is complete. If an error occurs during the Connect process, an error message is displayed in the Notification bar and the result column, and the Sequence window remains open.

If the instrument is already connected to the software, the Connect command is used to disconnect the instrument. A confirmation request dialog box is displayed when the command is selected, as shown below.

Figure 4.156 Disconnect Confirmation Request Dialog Box

Click the OK button to disconnect the instrument, or the Cancel button to abort the disconnect operation.

4.4.6.3 Standby

The Standby function is used to shut down the instrument. To access the Standby function, open a Sample Table and select Standby from the Instrument menu. The Standby window, which lists options for the shutdown operation, is displayed. Select the desired options and click the Standby button. Click the Escape button to abort the Standby operation and maintain the instrument in Ready mode.

Reference: *Refer to Section 4.3.4 "Ending Measurement" to end measurement.*

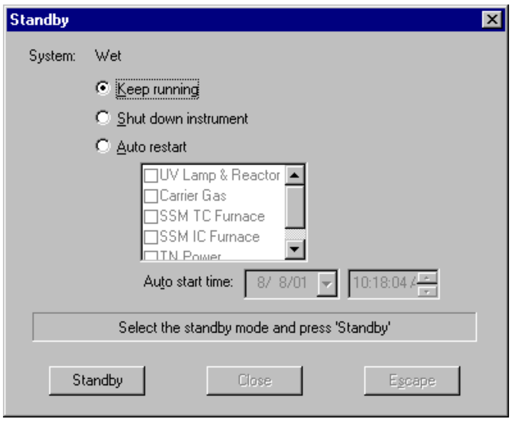


Figure 4.157 Standby Window

Item	Description
Shut down instrument	Select this option to begin instrument shutdown. After Execute is pressed, the carrier gas, UV lamp and TC reactor heater are turned off.
Auto restart	Select this option to shut down the instrument and automatically restart it at a specified time. Set the restart time using the spin controls for Auto start time. The default settings are the current system date and time.
UV Lamp & Reactor	Select this option to keep the TC reactor UV lamp and heater on for 30 minutes. This option is available only when the Auto restart option is selected.
Carrier Gas	Select this option to keep the carrier gas flowing for 30 minutes. This option is available only when the Auto restart option is selected.

4.4.6.4 Start(Continue)

The Start command is used to start the instrument. Start a sequence by opening a Sample Table, inserting samples, connecting the instrument, and selecting Start from the Instrument menu. Refer to Section 4.4.6.2 "Connect" for more information on connecting the instrument.

The Standby window is displayed. Click the Keep running radio button. If the ASI is installed, the Sparging/Acid Addition window is displayed. Verify the vial positions and click the Start button to begin measurement.

If the ASI is not installed, the Meas Mode dialog box is displayed. To perform continuous measurement or to continue measurement, click OK. Click cancel to abort the start procedure.

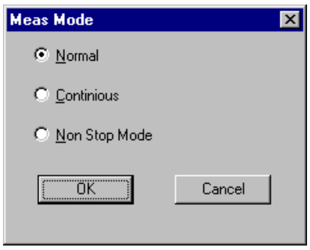


Figure 4.158 Meas Mode Dialog Box

Item	Description
Normal	This mode requires user input between each sample before the instrument will move to the next sample in the sample table.
Continuous	Select this mode to measure continuously through the sample table rows until the instrument is stopped by the user.
Non Stop Mode	Select this mode to perform measurement continuously using more than one type of measurement modes.

4.4.6.5 Stop>Peak Stop

The Stop>Peak Stop command is used to interrupt the current measurement. The peak measured up to the stopping point is considered a complete peak. After the Stop>Peak Stop command is executed, the next injection listed in the Sample Table is made and analysis continues normally.

4.4.6.6 Stop>Finish Current Sample

The Stop>Finish Current Sample command is used to interrupt measurement after all injections of the current sample are complete. After the Stop>Finish Current command is executed, the instrument remains in the Ready mode.

4.4.6.7 Stop>Halt

The Stop>Halt command is used to immediately interrupt the current measurement, discard data from the current injection, and return the instrument to the Ready mode.

4.4.7 Maintenance Menu

Instrument maintenance procedures are described in Section 5.5 "Software-Controlled Maintenance Functions" of this manual. All maintenance functions can be initiated using the options displayed in the Instrument>Maintenance menu.

4.4.8 Tools Menu

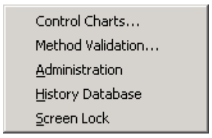


Figure 4.159

4.4.8.1 Administration

The Administration function is used to access the Administration module of the TOC-Control V software.

4.4.8.2 Screen Lock

If the user must temporarily leave the PC he/she is logged into, the screen can be locked by using the password function to prevent another individual from performing operations in TOC-Control V. This function can be used only if the User ID / Password item is checked in the Security dialog box.

To lock the screen, select Screen Lock in the Tools menu. The screen will then be locked and the Re-enter Password dialog box is displayed. To release the screen lock, enter the user ID and password into the designated fields. If the user ID and password entered are not the same as those used up to that point, the screen lock will not be released.

Figure 4.160 Re-enter Password Dialog Box

4.4.9 Options Menu

The Options menu contains commands for viewing and displaying system information and for setting default measurement parameters.

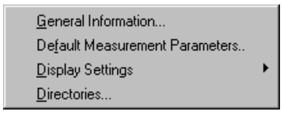


Figure 4.161 Options Menu

4.4.9.1 General Information

The General Information command is used to view information about the system. The General Information window contains two tabs, one that displays System parameters and one that displays History information. Most of the fields in the General Information window are disabled because they were set previously. After changing an active field, click the OK button to close the window and save the changes or click the Apply button to save the changes without closing the window. Click the Cancel button to close the window without saving changes.

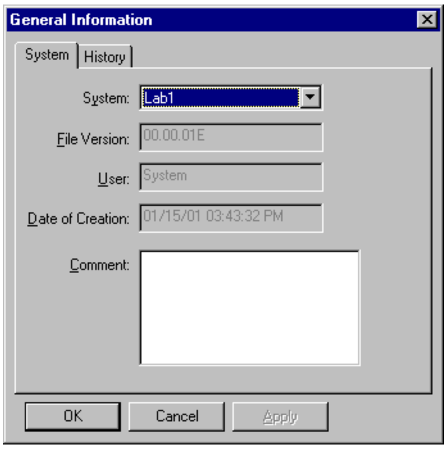


Figure 4.162 General Information Window: System Tab

System Tab

The System tab displays general parameters.

Parameter	Description
System	Displays the instrument identification.
File Version	Displays the instrument file version.
User	Displays the name of the user who set up the system.
Date of Creation	Displays the date the system was set up.
Comment	Enter a comment (512 characters maximum), if desired.

History Tab

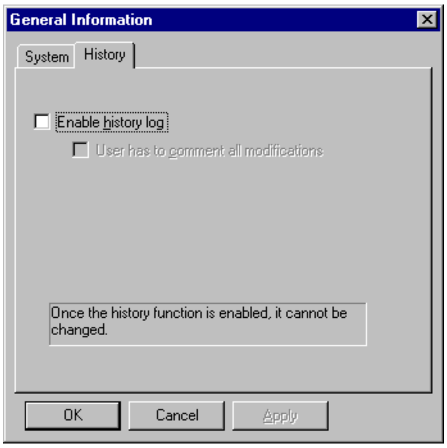


Figure 4.163 General Information Window: History Tab

The History tab is used to view the system's History Log or to enable the log if it was not previously selected.

Note: Once the History Log is enabled, it cannot be disabled.

Setting Item	Description
Enable history log	Select this option to enable the software to monitor all modifications to the system. If the History Log is not required, it need not be enabled, as the software operates properly without it.
User has to comment all modifications	Select this option to require comments or approval for all modifications.

4.4.9.2 Default Measurement Parameters

The Default Measurement Parameters command is used to enter the default values for samples, methods, and calibration curves. The Default Measurement Parameters window contains a separate tab for each analysis type, into which the default values are entered.

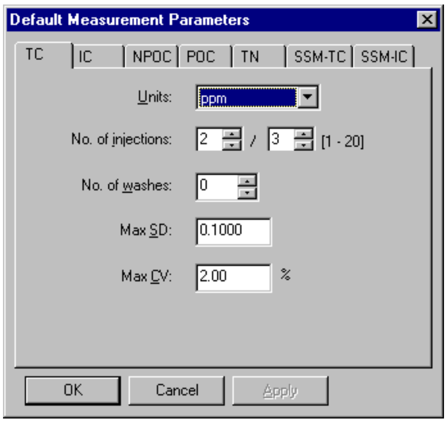


Figure 4.164 Default Measurement Parameters: TC/IC/NPOC/POC/TN/SSM-TC/SSM-IC Tabs

The parameters shown on each of the tabs are similar and are described below.

Parameter	Description
Units	Select the default concentration units.
No. of injections	Enter the default minimum/maximum number of injections.
No. of washes	Enter the default number of times the syringe is washed with sample prior to the first injection. If the Auto Dilution option is selected, the sample is diluted and the syringe washed with the diluted sample.
Max SD	Enter the default maximum acceptable standard deviation. If this value is met, no additional injections are required. If both this value and the Max CV value are exceeded, the samples are automatically reinjected up to the maximum number of times. The SD Max field is disabled if the No. of Injections is 1 or if the minimum is equal to the maximum.
Max CV	Enter the default maximum acceptable coefficient of variation. If this value is met, no additional injections are required. If both this value and the Max SD value are exceeded, the samples are automatically reinjected up to the maximum number of times. The CV Max field is disabled if the No. of Injections is 1 or if the minimum is equal to the maximum.

4.4.9.3 Display Settings>Display Font

The Display Settings>Display Font function is used to select font options for both the general screen display (such as in the Sample Table and Print Preview screens) and printed reports. Select a font type, style, size, and color from the lists in the Font dialog box. A preview of the font appearance is displayed in the Sample field.

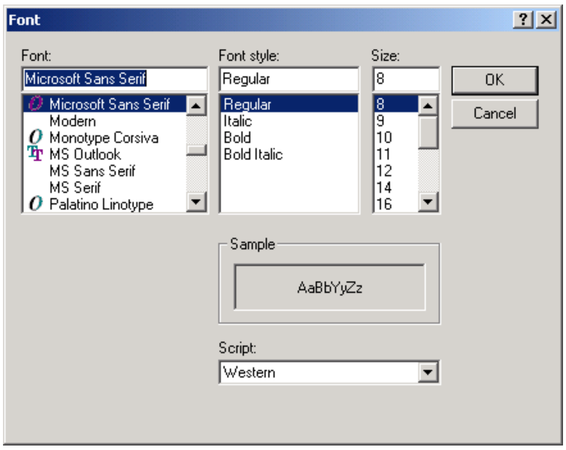


Figure 4.165 Font Dialog Box

4.4.9.4 Display Settings>Table Settings

The Display Settings>Table Settings function is used to select the parameters that are displayed in the Sample Table window. The Table Options window contains two tabs, one that lists Sample Table parameters and one that lists Injection Table parameters. On each tab, certain options are selected by default. Individual options can be selected or deselected by clicking in the check box. Use the Select All and Select None buttons to quickly select and deselect all options on a tab. Use the Default button to select the default options.

Sample Table Tab

Select the parameters that are displayed in the Sample Table.

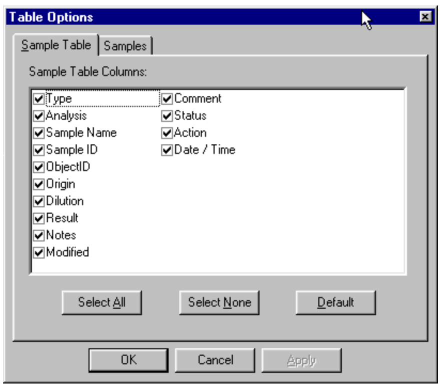


Figure 4.166 Table Options Window: Sample Table Tab

Samples Tab

Select the parameters that are displayed in the Injection Table.

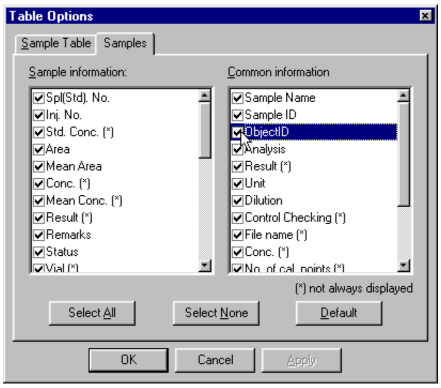


Figure 4.167 Table Options Window: Samples Tab

4.4.9.5 Display Settings>Notification Bar Settings

The Display Settings>Notification Bar Settings function is used to set options for the Notification bar, where error messages and other notices are displayed. The Notification bar is located at the bottom of the Sample Table Editor window.

Four folders can be displayed in the Notification Bar: Notifications, System Folders, Errors, and Events. When a folder is selected, a tab appears for that folder in the Notification bar. When more than one folder is selected for display, the contents of a specific folder can be displayed by clicking the appropriate tab.

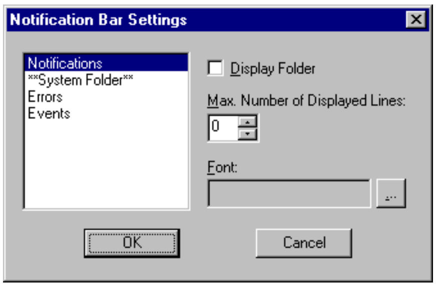


Figure 4.168 Notification Bar Settings Window

Setting Item	Description
Folder List	Lists the folders that can be displayed in the Notification bar.
Display Folder	Place a check in this box to display the folder.
Max. Number of Displayed Lines	Enter the maximum number of lines to be displayed in the Notification bar. Acceptable values range from 0 to 200.
Font	Displays the selected font. Modify the font by selecting the Browse button to open the Font dialog box.

To select a folder for display in the Notification bar, click the folder in the Folder List, then select the Display Folder option. Set the Max. Number of Displayed Lines field to a value greater than 0. To change the color or appearance of the text displayed in the folder, use the Font option. The Display Folder check box, Max. Number of Displayed Lines field, and Font option apply only to the selected folder; as a result, these options must be set separately for each folder.

Note: A folder is not displayed in the Notification bar unless the Display Folder option is selected and the Max. Number of Displayed Lines field is set to a value greater than 0.

4.4.9.6 Display Settings>Floating Point Number Formats

The Display Settings>Floating Point Number Formats command is used to select display options for numerical results in the Sample Table. These options apply only to the manner in which numbers are displayed; internal data retain their original values.

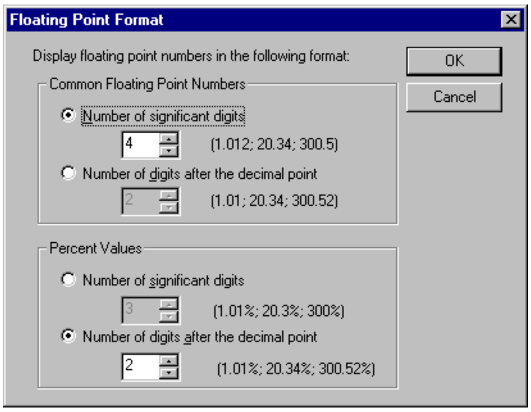


Figure 4.169 Floating Point Format Window

Select settings for both common numbers and percent values, as described below.

Common Floating Point Numbers

Setting Item	Description
Number of significant digits	Select this option to display common floating point numbers using a specified number of significant digits. Use the spin control to set the number of significant digits. The default value is 4.
Number of digits after the decimal point	Select this option to display common floating point numbers using a specified number of digits after the decimal point. Use the spin control to set the number of digits after the decimal point. The default value is 2.

Percent Values

Setting Item	Description
Number of significant digits	Select this option to display percent values using a specified number of significant digits. Use the spin control to set the number of significant digits. The default value is 4.
Number of digits after the decimal point	Select this option to display percent values using a specified number of digits after the decimal point. Use the spin control to set the number of digits after the decimal point. The default value is 2.

4.4.9.7 Directories

The Directories function is used to modify system directories. Select Options>Directories to display the Folder window.

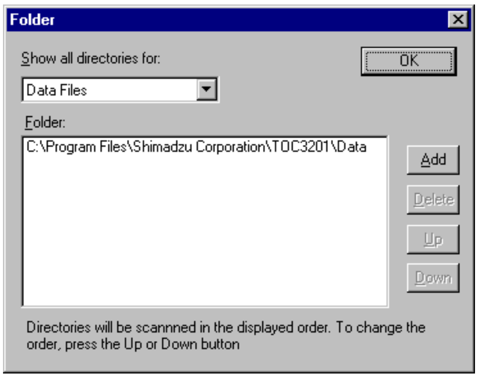


Figure 4.170 Folder Window

Show all directories for

Use the drop-down list to access various directories. Available directories are listed in the Folder text field.

Add

Click the Add button to add a new directory to the list for a specified topic, and the Folder dialog box is displayed. Highlight the desired directory from the drop-down Drive and Directory lists, then select it by clicking the OK button. Click the Network button to access a network drive.

Figure 4.171 Folder Dialog Box

Delete

Click the Delete button to delete a selected directory from the list.

Up

Click the Up button to move the selected directory up one line in the list. The order of the directories in the list determines the order directories are searched during instrument operation.

Down

Click the Down button to move the selected directory down one line in the list. The order of the directories in the list determines the order directories are searched during instrument operation.

4.4.10 Window Menu

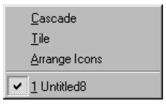


Figure 4.172 Window Menu

Cascade

The Cascade command is used to stack all open windows so that the title bars are visible.

Tile

The Tile command is used to size all open windows so that each window occupies an equal portion of the screen.

Arrange Icons

The Arrange Icons command is used to neatly organize windows that have been minimized or reduced to icons.

4.4.11 Help Menu

Figure 4.173 Help Menu

About

The About command is used to view copyright, software registration, and software version information.

5 Maintenance

Inspection and maintenance of the TOC-V instrument are required to ensure normal operation and reliable and accurate data acquisition.

5.1 Daily Inspection

Check the levels of dilution water, oxidizing reagent, acid and drain vessel water before starting the instrument for the day's operations.

5.1.1 Checking the Dilution Water

Verify that there is sufficient dilution water for analysis. Referring to Section 4.1.8 “Preparation of Dilution Water”, replenish the dilution water as necessary. To obtain good measurement reproducibility, the dilution water should be replaced with fresh purified water every day.

5.1.2 Checking the Persulfate Oxidizer Reagent

Check the oxidizer before using the instrument. Referring to Section 4.1.6 “Preparing the Persulfate Oxidizer Solution”, replenish or replace the persulfate oxidizer reagent as necessary. Depending on the amount normally used, a narrow-mouth 1L glass bottle can be used as a suitable container.

5.1.3 Checking the Acid

Verify that there is adequate acid for analysis. Referring to Section 4.1.7 “Preparing Acid”, replenish or replace the acid as necessary. Depending on the amount normally used, a narrow-mouth 1L glass bottle is a suitable container.

5.1.4 Checking the Drain Vessel Water Level

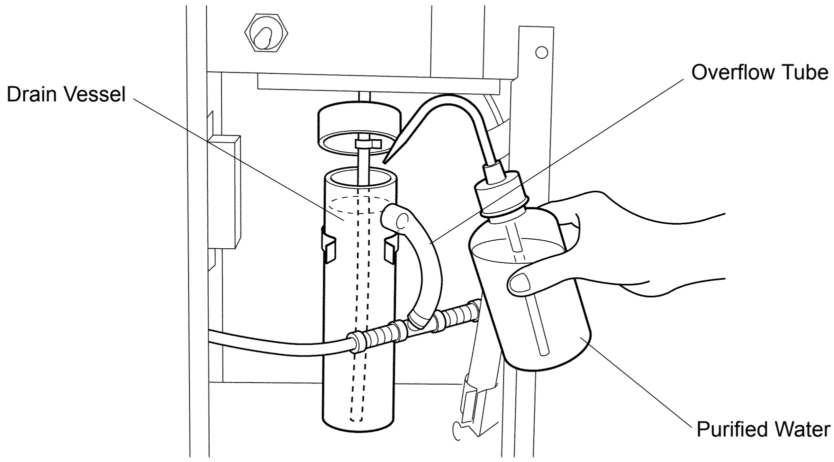


Figure 5.1 Drain Vessel Water Level

Verify that the water level in the drain vessel inside the instrument is within 10mm of the overflow tube on the side of the drain vessel. If the level is lower replenish using purified water.

Confirm that carrier gas does not leak from the tip of the drain tubing when the flow rate is 200mL/min.

Reference: *Refer to Section 4.1.5 "Water Supply to the Dehumidifier Drain Container" for details on adding purified water.*

5.1.5 Checking the Waste Container

Before using the instrument, check the liquid level in the waste container. Since the waste liquid is corrosive (acidic), ensure that the remaining container capacity is sufficient to prevent overflow of the container during instrument operation. Empty the container if there is insufficient capacity.

5.2 Periodic Inspections

To maintain the performance of the instrument, perform periodic checks of the items listed in this section.

5.2.1 Replacing the High Purity Nitrogen (Cylinder)

A 47L cylinder of high purity nitrogen carrier gas will last about 1.5 months. Replace the cylinder before it is completely empty, while the remaining pressure is still several hundred kPa. When connecting the new cylinder, clean the connection port, and after connecting the tubing, be careful not to allow any dust to enter the instrument.

Keep one full cylinder available as a spare.

5.2.2 Replacing the CO_2 Absorber

Once a year, replace each of the CO_2 absorbers (P/N 630-00999) installed behind the instrument.

CO_2 Absorber Disposal Procedure

Used CO_2 absorbers should be disposed of according to local industrial waste disposal regulations. It must be clearly disclosed using an industrial waste manifest that the CO_2 absorber contains soda lime.

Reference: *Refer to Section 4.1.9 "Installation of the CO_2 Absorber" for details on replacing the CO_2 absorbers.*

5.2.3 Replacing the Halogen Scrubber

The absorbent inside the halogen scrubber turns black as it absorbs chlorine; this discoloration band advances toward the outlet as the amount of chlorine absorbed increases. When the front of the discoloration band reaches the position shown in Figure 5.2 "Halogen Scrubber", replace the scrubber with a new halogen scrubber.

Replacement Procedure

1. Open the upper and lower clips securing the halogen scrubber.
2. Remove the upper and lower connection clamps.
3. Disconnecting the Viton connectors remove the halogen scrubber.

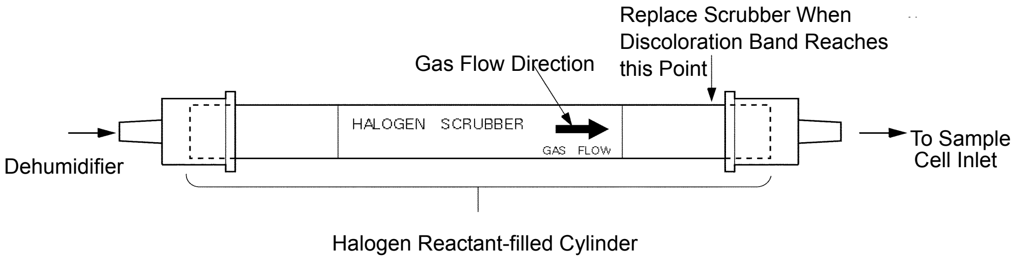


Figure 5.2 Halogen Scrubber

Note: The halogen scrubber protects the more expensive non-dispersive infrared (NDIR) detector from corrosion. The halogen scrubber should be replaced as described above to avoid damage to the NDIR.

The baseline may fluctuate after replacement. Allow the instrument to run for a short time until the baseline stabilizes. Stabilization normally occurs within one hour.

Used halogen scrubbers should be disposed of according to local industrial waste disposal regulations. It must be clearly disclosed using an industrial waste manifest that the halogen scrubber contains copper

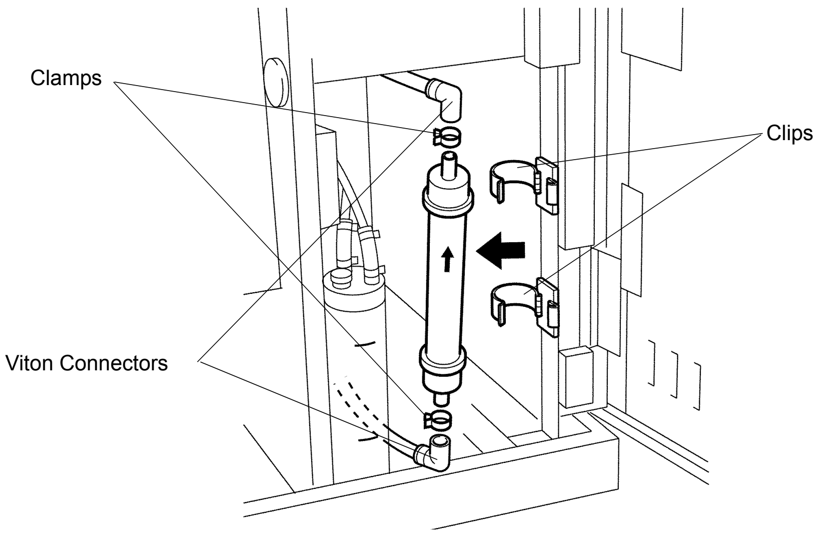


Figure 5.3 Halogen Scrubber Replacement

5.2.4 Replacing the Syringe Plunger Tip

The syringe plunger tip is constructed of fluororesin and wears with use. Gaps will eventually form between the plunger and the inner wall of the syringe barrel (glass tube) causing leaks. When leaks occur, bubbles are produced near the syringe tip while the sample is being drawn into the syringe or the sample may leak from the bottom of the barrel when the sample is discharged. Replace the plunger tip if either of these situations occur.

Replacement Procedure

1. Refer to Section 4.1.3 "Installing the Syringe" to remove the syringe.

2. As shown in Figure 5.4 "Removing the Old Plunger Tip", use a sharp knife to make two or three incisions at the bottom of the plunger tip.

3. Grasp the plunger tip with a pair of pliers and remove it from the plunger. The O-ring that is held in place by the plunger tip also comes off. DO NOT reuse this O-ring.

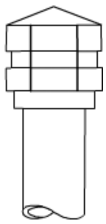


Figure 5.4 Removing the Old Plunger Tip

4. Refer to Figure 5.5 "Installing the New Plunger Tip (Cross-section)". Push down on the plunger, press the leading edge of the new plunger tip, straight into the plunger tip seat.

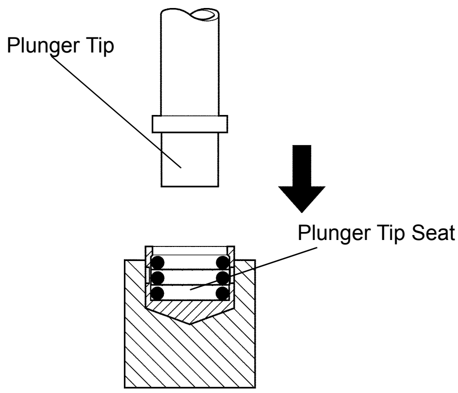


Figure 5.5 Installing the New Plunger Tip (Cross-section)

Note: Do not to scratch the perimeter of the plunger tip. This could cause leaks. Use only Shimadzu-specified plunger tips. Use of unspecified parts may result in instrument malfunction and/or diminished performance.

5.2.5 Washing the TC Reactor

Procedure

1. Loosen both of the TC reactor cover screws.

2. Remove the 6 retaining pins that secure the TC reactor heater insulation, and open both of the TC reactor heater retaining brackets.

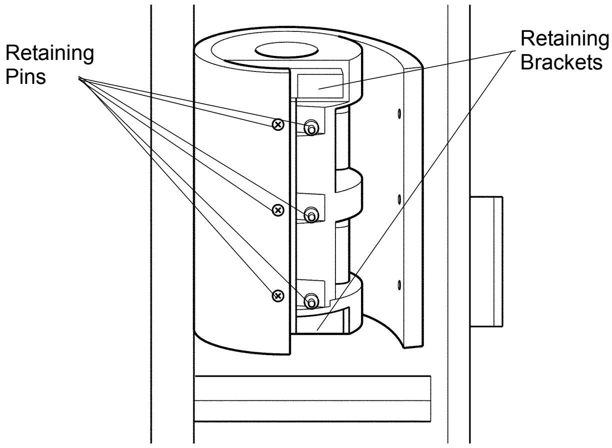


Figure 5.6 TC Reactor Heater

3. Remove the TC reactor, and disconnect the tubing from the narrow end and branch tube of the TC reactor.

4. Open the upper door, and detach the UV lamp connector from the UV lamp power supply board.

5. Referring to Figure 5.7 “Removing the UV Lamp”, pull the UV lamp out of the TC reactor along with the TC reactor cap.

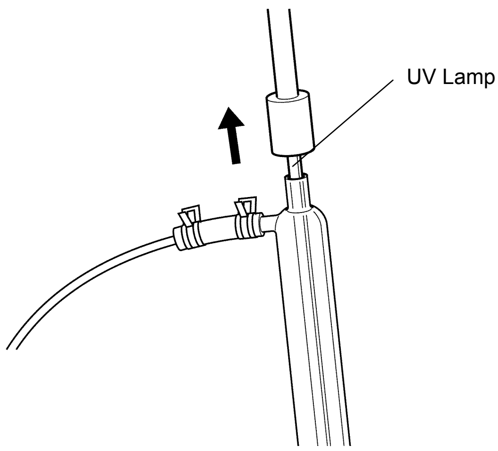


Figure 5.7 Removing the UV Lamp

6. Wash the TC reactor and UV lamp and rinse well with purified water. Do not allow water to contact the UV lamp lead or connector.

7. Using the procedure described in Section 4.1.1 "Installing the TC reactor", re-install the TC reactor and UV lamp.

CAUTION

- Turn OFF the TC reactor heater and the UV lamp. Wait for the heater to cool to room temperature before performing any maintenance on the TC reactor. There is a danger of burn injury and electric shock.
- High voltage is applied to the UV lamp and UV lamp power supply. Due to the danger of electric shock, turn OFF the UV lamp before performing any maintenance on the TC reactor.
- The oxidizing reagent is corrosive and may leak into the TC reactor. DO NOT spill or touch this reagent.
- The TC reactor is glass. To avoid injury, do not break the glass when connecting the TC reactor.
- When tightening the plastic nuts used on the connectors, do not use any tools. Tightening securely by hand is sufficient.
- Do not contaminate the internal or external surfaces of the TC reactor or its connections with organic substances such as grease or oil. This is critical for high sensitivity measurement.

5.2.6 Washing the IC Reactor

Procedure

1. Remove the IC reactor from the mounting clamps.

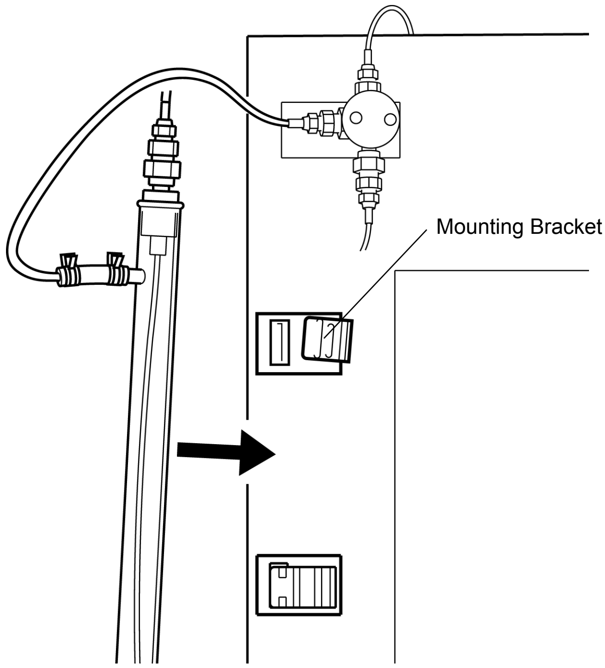


Figure 5.8 Removing the IC Reactor

2. Disconnect the tubing from the narrow end and branch tube.

3. Remove the rubber cap from the top of the IC reactor along with the sparge tubing.

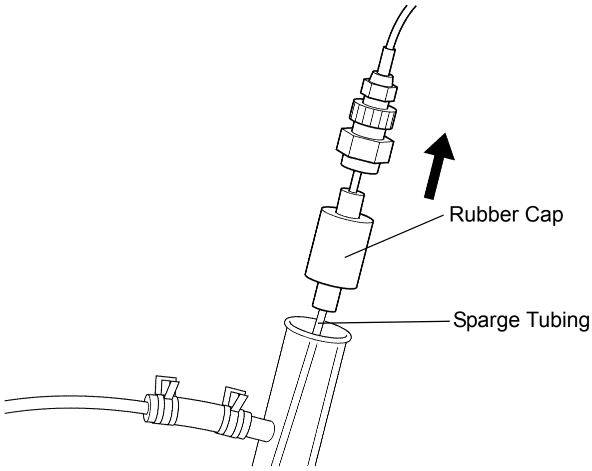


Figure 5.9 Removing the Rubber Cap

4. Wash the IC reactor and rinse it well with purified water.

5. Using the procedure described in Section 4.1.2 "Installing the IC Reactor", re-install the IC reactor.

CAUTION

- Phosphoric acid is a corrosive substance and may leak inside the IC reactor. DO NOT spill or touch this substance.
- The IC reactor is glass. To avoid injury, do not break the glass when installing the IC reactor.
- When tightening the plastic nuts used on the connectors, do not use any tools. Tightening securely by hand is sufficient.
- Do not contaminate the internal or external surfaces of the IC reactor or its connections with organic substances such as grease or oil. This is critical for high sensitivity measurement.

5.2.7 Replacing the UV Lamp

Replace the UV lamp once a year

Procedure

1. Using the procedure described in Section 5.2.5 “Washing the TC Reactor”, remove the UV lamp.

2. Using the procedure described in Section 4.1.1 “Installing the TC reactor”. install a new UV lamp.

CAUTION

- Turn OFF the TC reactor heater and the UV lamp. Wait for the heater to cool to room temperature before performing maintenance on the TC reactor. There is a danger of burn injury and electric shock.
- High voltage is applied to the UV lamp and UV lamp power supply. Due to the danger of electric shock, turn OFF the UV lamp before performing maintenance on the TC reactor.
- The oxidizing reagent is corrosive and may leak into the TC reactor. DO NOT spill or touch this reagent.
- The TC reactor is glass. To avoid injury, do not break the glass when connecting the TC reactor.
- When tightening the plastic nuts used on the connectors, do not use any tools. Tightening securely by hand is sufficient.
- Do not contaminate the internal or external surfaces of the TC reactor or its connections with organic substances such as grease or oil. This is critical for high sensitivity measurement.

5.2.8 Replacing the Syringe Pump 8-Port Valve Rotor

The fluororesin rotor of the syringe pump 8-port valve gradually becomes worn and scratched after prolonged use, due to suspended particles in the samples. This wear eventually causes leaks from the 8-port valve.

The following symptoms are associated with leaks from the 8-port valve and indicate that it is necessary to replace the rotor.

- Droplets of liquid around the 8-port valve (around the upper shaft)
- Decreased analysis reproducibility
- Presence of bubbles in the syringe

If there is a large leak, sample remaining in the tubing after the syringe has been filled may leak back into the sample container. The sample tubing flange connection may be the source of this problem.

CAUTION

Switch OFF the instrument before replacing the 8-port valve rotor.

Procedure

1. Turn OFF the power to shut down the instrument.

2. Verify that the **O** mark on the 8-port valve interrupt plate is directed toward the No. 1 port. If it is not, the instrument was not shut down using the appropriate procedure. If this is the case, switch the instrument back ON, and follow the correct shutdown procedure.

Reference: *Refer to Section 4.3.4 "Ending Measurement" for the correct shutdown procedure.*

3. Remove the nut using the provided wrench.

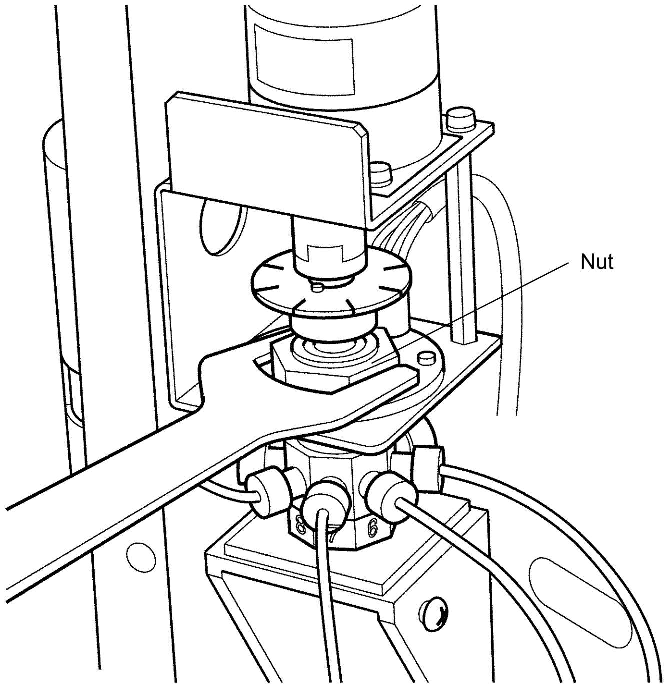


Figure 5.10 Removing the Nut

4. Pull up to remove the rotor drive component. Hang it on the screw on the right side of the syringe assembly housing.

5. Loosen the retaining nut using the provided wrench.

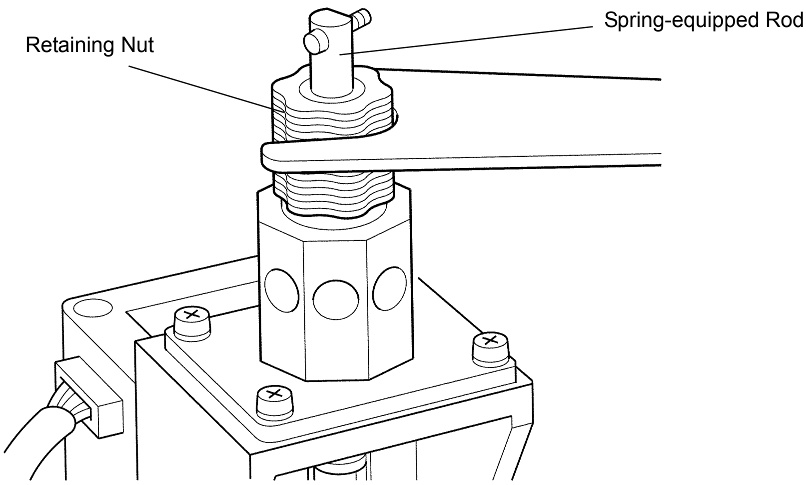


Figure 5.11 Removing the Retaining Nut

6. Take notice of the orientation (toward port No. 1) of the flat surface of the spring-equipped rod. Remove the spring-equipped rod and retaining nut.

7. Take notice of the orientation of the rotor hole position. Grasp the rotor with forceps, and remove it by pulling upwards.

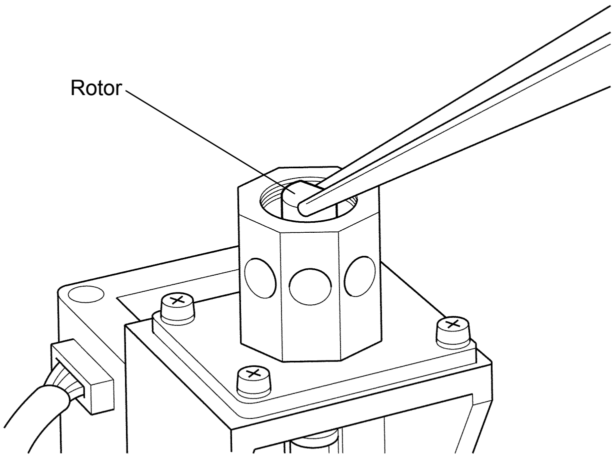


Figure 5.12 Removing the Rotor

8. Insert a new rotor, taking care that the rotor hole matches original orientation.

9. Reinsert the retaining nut and spring-equipped rod, with the flat surface directed toward the No. 1 port. Tighten the retaining nut.

10. Replace the rotor drive component, and tighten the nut.

11. Switch the instrument ON.

Reference: *Perform the syringe pump zero detection procedure before analysis. Refer to Section 4.1.4 "Syringe Pump Zero Point Detection" for more information.*

5.3 ASI-V Autosampler Maintenance

Maintenance of the autosampler includes periodic inspection of the rinse bottle, sample catcher and rinse pump.

5.3.1 Rinse Bottle Inspection

Perform the following inspections before conducting analysis with the autosampler. If the following inspections are not properly conducted, air may be drawn into the autosampler injection pump, preventing the delivery of rinse water.

Check the Rinse Water Level

Verify that the rinse water in the rinse bottle is above the 2-liter mark. Add water if it is below this level.

Check the Rinse Tubing

Verify that the tip of the rinse tubing reaches nearly to the bottom of the rinse bottle. Adjust the tubing depth if necessary.

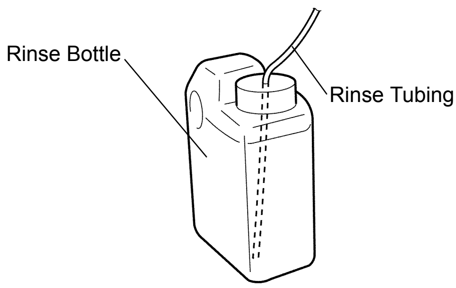


Figure 5.13 Rinse Bottle Inspection

5.3.2 Sample Catcher Inspection

Examine the turntable (black plate) and the sample catcher tray below the turntable when installing or removing the autosampler vial rack. If they are wet or dirty, wipe them clean.

CAUTION

Corrosion of the instrument may occur if spilled sample is allowed to remain in the sample catcher. Keep the sample catcher clean.

5.3.3 Replacing the Pump Head of the Rinse Pump

The rinse pump is located inside the autosampler and supplies rinse water to the rinse port.

The rinse pump is a peristaltic pump where a rotating roller flattens the rubber tubing that carries the rinse water. This rubber tubing gradually deteriorates requiring periodic replacement of the entire pump head.

The life of the pump head is generally about 300 hours of pump operation.

Replacement Procedure

Use the following procedure to replace the pump head.

1. Remove the rectangular pump access cover on the back left side of the autosampler.

2. Grasp the latch on the pump head by hand and pull the white pump head from the black pump body.

3. On the left side of the pump head, disconnect both of the yellow rubber tubes from the Teflon tubes.

4. Connect the new pump head tubing to the Teflon tubes.

5. Replace the pump head and the replace the pump access cover.

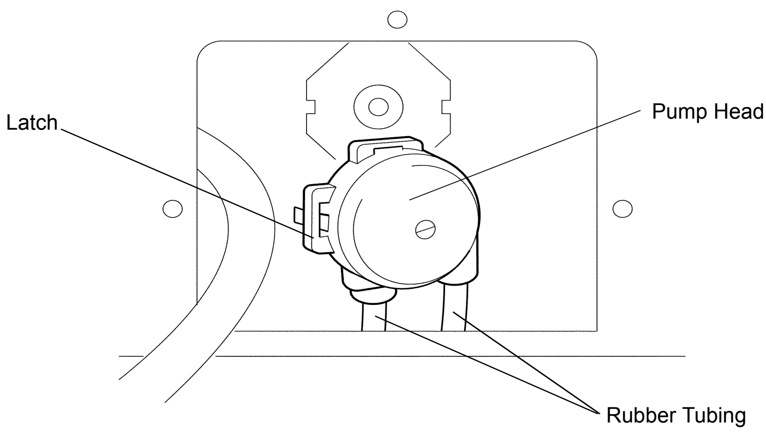


Figure 5.14 Replacing the Pump Head of the Rinse Pump

5.4 8-Port Sampler (OCT-1) Maintenance

5.4.1 Replacing the 8-Port Valve Rotor

The OCT-1 uses a fluororesin 8-port valve rotor. The valve rotor may become worn or scratched after prolonged use due to the suspended particles in the samples. This wear eventually causes leaks from the 8-port valve.

The following symptoms are associated with leaks from the 8-port valve and indicate that it is necessary to replace the rotor.

- Droplets of liquid around the 8-port valve (around the upper shaft)
- Decreased analysis reproducibility.
- Presence of bubbles in the TOC-V syringe.

If the leak is large, sample remaining in the tubing after the syringe has been filled may flow back into the sample container. This is most likely due to a sample tubing or flange connection problem.

⚠ CAUTION

Switch OFF the power before replacing to the 8-port valve rotor.

Replacement Procedure

1. Shut down the TOC-V unit and switch OFF the power.

Reference: *Refer to Section 4.3.4 "Ending Measurement" for details on shutting down the TOC-V unit.*

2. Remove the 4 screws at the top of the 8-Port Sampler, and take OFF the cover.

3. Verify that the **O** mark on the 8-port valve cover is directed toward port No.1.

   Note: If the **O** mark is not directed toward port No.1, the instrument was not shut down properly. If this is the case, turn the TOC-V on again, and shut it down properly.

4. Remove the nut using the wrench supplied with the TOC-V.

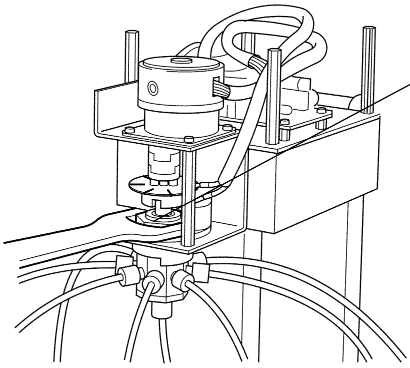


Figure 5.15 Removing the Nut

5. Remove the 8-port valve by pulling downward.

6. Loosen the retaining nut using the provided wrench.

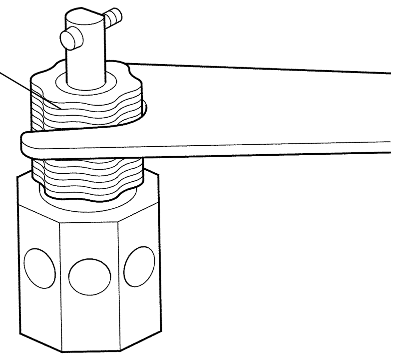


Figure 5.16 Removing the Retaining Bracket

7. Note the orientation (toward port No. 1) of the flat surface of the spring-equipped rod. Remove the spring-equipped rod and retaining nut.

8. Note the orientation of the rotor hole position. Grasp the rotor with forceps, and remove by pulling upwards.

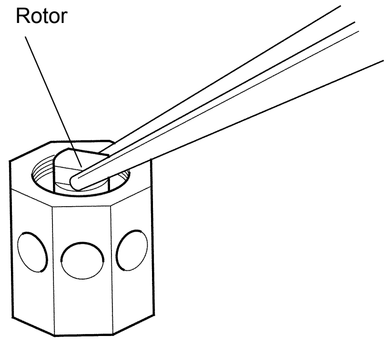


Figure 5.17 Removing the Rotor

9. Insert a new rotor, positioning the rotor hole to the original position.

10. Reinsert the spring-equipped rod and retaining nut with the flat surface directed toward port No. 1. Tighten the retaining nut.

11. Replace the 8-port valve in its original position, and secure it using the nut.

12. Replace the cover of the 8-Port Sampler and secure it with four screws.

13. Switch on the TOC-V unit.

5.5 Software-Controlled Maintenance Functions

Maintenance functions that can be performed using the TOC-Control V software are described below. These functions are accessed through the Instrument>Maintenance menu:

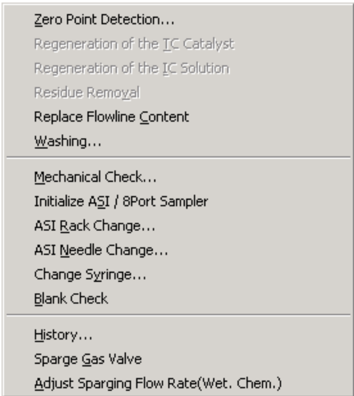


Figure 5.18 Instrument>Maintenance Menu

To access the menu, select Instrument>Maintenance in the Sample Table Editor.

5.5.1 Zero Point Detection

Use this function to determine the zero point of the syringe. Click the Start button to begin the procedure, during which a progress bar is active. The progress bar becomes inactive when the syringe zero point is determined. Click the Close button.

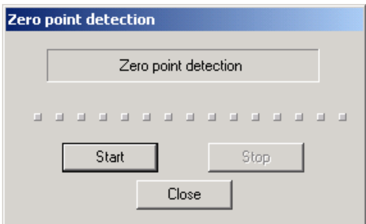


Figure 5.19 Zero Point Detection Dialog Box

5.5.2 Replace Flowline Content

Select this command to remove bubbles from the dilution solution tubing, the oxidizer tubing or the acid tubing. This should be executed immediately after instrument installation, when reagents are replenished, or whenever bubbles are present in the tubing.

To start bubble removal, click the “Start” button.

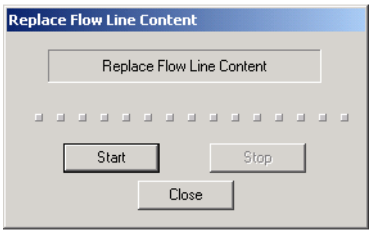


Figure 5.20 Replace Flowline Content Dialog Box

5.5.3 Washing

Use this function to wash selected flow lines.

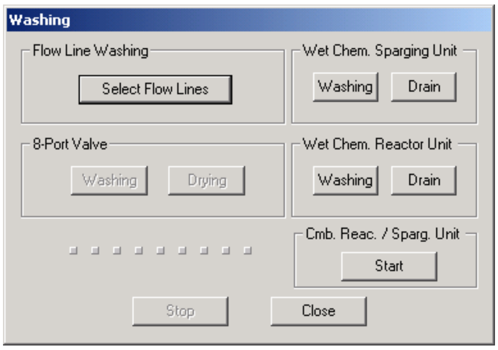


Figure 5.21 Washing Dialog Box

To perform flow line washing, click the Select Flow Lines button to open the Wash Flow Lines dialog box. Select the flow lines to be washed, and click the Wash button. The ports for the 8-Port Sampler 1 (2) can be selected if the 8-Port Sampler 1 (2) is being used.

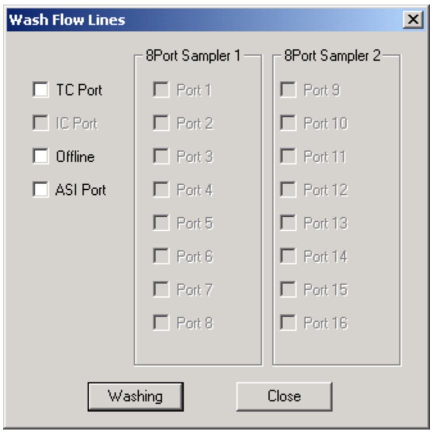


Figure 5.22 Wash Flow Lines Dialog Box

While this process is being executed, the buttons in the Washing dialog box are disabled, and the progress bar activity is displayed.

Flow Line Washing [TC]	Washes the sample tubing.
8-Port Valve [Washing]	Washes the inside of the 8-port valve.
8-Port Valve [Drying]	Dries the inside of the 8-port valve.
Wet Chem. Sparging Unit [Washing]	Washes the inside of the IC reactor with dilution water.
Wet Chem. Sparging Unit [Drain]	Expels the contents of the IC reactor.
Wet Chem. Reactor Unit [Washing]	Washes the inside of the TC reactor with dilution water.
Wet Chem.Reactor Unit [Drain]	Expels the contents of the TC reactor.
Cmb. Reac. / Sparg. Unit	Washes the inside of the TC and IC reactor using dilution water.

5.5.4 Mechanical Check

Select this function to check the mechanical components of the instrument or to make adjustments. The Mechanical Check window consists of several tabs, one for each connected instrument.

TOC Tab

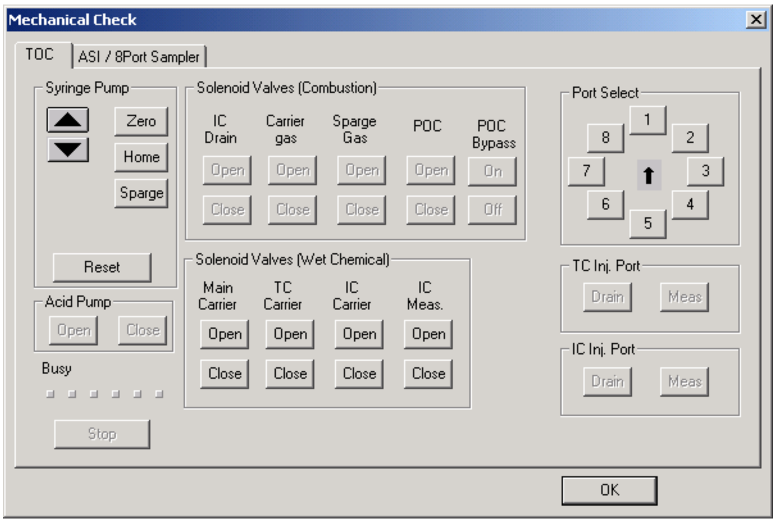


Figure 5.23 Mechanical Check Window: TOC Tab

Syringe Pump

Use the buttons to move the syringe pump to the Zero, Home, or Sparge positions. Click the Reset button to move the plunger to the highest position.

Solenoid Valves

Use the buttons to open or close the various solenoid valves. Different options are available depending on the instrument configuration and oxidation method.

Port Select

Open the line between the syringe and an injection port by clicking the button for the desired port.

ASI / 8-Port Sampler Tab

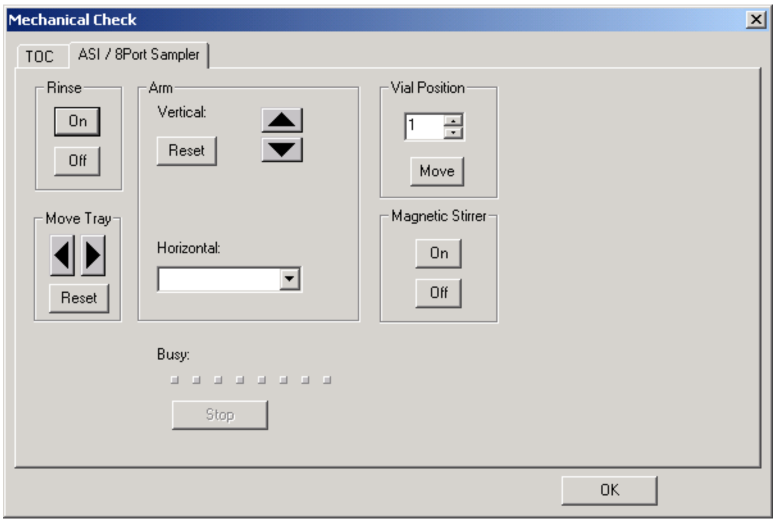


Figure 5.24 Mechanical Check Window: ASI Tab

Rinse

Use the On and Off buttons to start and stop the ASI flow line rinsing option.

Move Tray

Use the directional arrows to move the ASI tray to the desired position. Click the Reset button to return the tray to its home position.

Arm

Use the directional arrows to move the arm vertically. Use the drop-down list to move the arm horizontally.

Vial Position

Use the spin controls to enter a vial position, and then click the Move button to verify that the arm moves to the specified vial position.

5.5.5 ASI / 8-Port Sampler Initialization

Use this function when first connecting the instrument to establish communication between the TOC-Control V software and the ASI-V autosampler or the OCT-1 8-Port Sampler. This option is disabled if an ASI or 8-Port Sampler is not connected to the system.

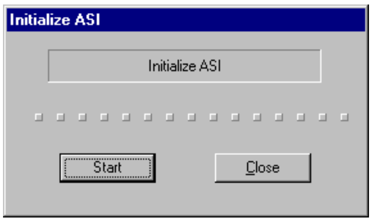


Figure 5.25 Initialize ASI / 8-Port Sampler Window

Click the Start button to begin the procedure and activate the progress bar. The progress bar becomes inactive when the ASI or 8-Port Sampler is initialized. Click the Close button.

5.5.6 ASI Rack Change

Use this function to change the ASI rack. This option is disabled if an ASI is not connected to the system.

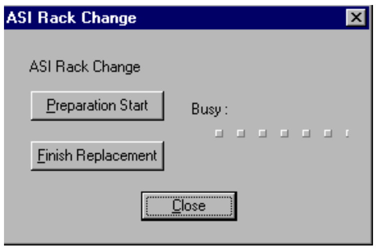


Figure 5.26 ASI Rack Change Window

Click the Preparation Start button to begin the procedure. A progress bar is active while the rack moves to the position where it can be replaced. When the progress bar begins blinking, replace the rack. Click the Finish Replacement button to initialize the ASI and complete the rack change process. Click the Close button.

5.5.7 ASI Needle Change

Use this function to change the ASI needle. This option is disabled if an ASI is not connected to the system.

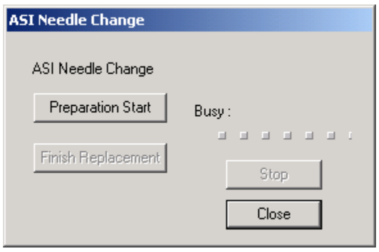


Figure 5.27 ASI Needle Change Window

Click the Preparation Start button to begin the procedure. A progress bar is active while the needle moves to the position where it can be replaced. When the progress bar begins blinking, replace the needle. Click the Finish Replacement button to initialize the ASI and complete the needle change process. Click the Close button.

5.5.8 Change Syringe

Use this function to replace the syringe.

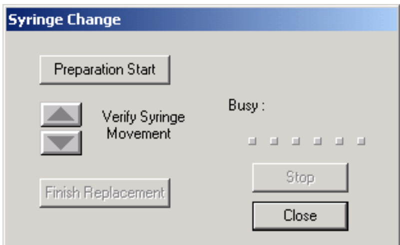


Figure 5.28 Syringe Change Window

Click the Preparation Start button to begin the procedure. A progress bar is active while the syringe moves to the position where it can be replaced. When the progress bar begins blinking, replace the syringe. Verify that the syringe moves smoothly between the upper and lower positions by clicking the directional arrows. Click the Finish Replacement button to move the syringe to its initialization position, then click the Close button.

Note: Perform the zero point detection procedure whenever the syringe is newly installed or the syringe is replaced. Refer to Section 4.1.4 "Syringe Pump Zero Point Detection" for details.

5.5.9 Blank Check

Use this function to automatically determine the background TC level of the instrument. Conducting the Blank Check procedure is recommended prior to performing high sensitivity measurements. Open a blank Sample Table, connect the instrument, and select the Blank Check option from the Instrument menu. Refer to Section 4.3.3 “TC Blank Check Analysis” for details. Set the injection volume using the spin controls, then click the Start button to begin the procedure.

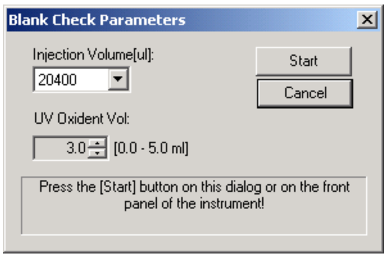


Figure 5.29 Blank Check Parameters Window

Note: Do not insert a sample into the Sample Table. The Blank Check procedure only works in a blank Sample Table.

5.5.10 History

Use this option to view the operational history of various instrument components and to set limits for various parameters of the components. When a limit is exceeded for an instrument component, a warning message is generated on the PC screen to remind the user to perform maintenance on that component. Refer to Section 4.2.4 “Maintenance History Settings” for a detailed description of this function.

5.5.11 Sparge Gas Valve

Select this option to start and stop the flow of sparge gas when the external sparge kit is used.

5.5.12 Adjusting Sparge Gas Flow Rate (Wet Chem.)

Use this function to adjust the flow of sparge gas to the IC reactor. Refer to Section 4.3.1.4 “Setting Sparge Gas Flow Rate” for details.

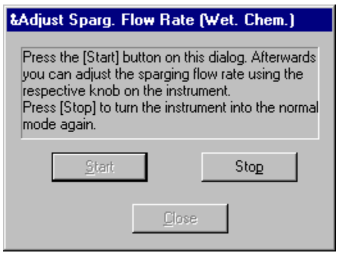


Figure 5.30 Adjusting Sparge Flow Rate (Wet Chem)

5.6 Troubleshooting

This section describes the error messages displayed by the TOC-Control V software and the actions recommended to resolve the underlying causes.

5.6.1 Error Messages

The following error messages are displayed on the PC screen when setting or operation errors are made, or when problems with instrument operation occur. Take the corrective actions indicated in the table below.

Table 5.1 Error Message List 1

Error Message	Description	Corrective Action
WARNING: Dilution water depleted	Actual amount of dilution water used exceeds value set in the Maintenance History Settings.	Replenish the dilution water. Then, reset the Dilution Water amount in the Maintenance History Settings.
WARNING: Acid depleted	Actual amount of phosphoric acid used exceeds value set in the Maintenance History Settings.	Replenish the phosphoric acid. Then, reset the Total Acid Volume in the Maintenance History Settings.
WARNING: Oxidizer Volume	The volume of oxidizer used exceeded the value set in the Maintenance Log Settings.	Replenish the oxidation solution. Reset "Wet Chemical Oxidizer Usage Volume" in the Maintenance Log Settings.
ERROR: Syringe stroke number exceeded	Actual number of syringe strokes performed exceeds value set in the Maintenance History Settings.	Replace the plunger tip. Then, reset the Syringe Stroke value in the Maintenance History Settings.
WARNING: ASI pump tube operating hours exceeded.	Actual ASI pump tubing operation time exceeds value set in the Maintenance History Settings.	Replace the ASI pump tubing. Then, reset the ASI Rinse Pump time in the Maintenance History Settings.

Table 5.2• Error Message List 2

Error Message	Description	Corrective Action
Information entered is incorrect. Re-enter user name and password.	The user name and password entered are not valid.	Enter the correct user name and password. These fields are case-sensitive. If the user account has not been set up, contact the system administrator.
Database path not found.	The specified file does not exist in the specified path.	Verify the file name and path. Determine if the specified file has been deleted or moved, or if the system used for the file has been deleted.
Opening of communication port has timed out. Check instrument connection and transmission settings.	The process of opening the communication port has timed out.	Verify that the instrument is switched On, and that the instrument connection and communication port settings are correct.
Closing of communication port has timed out. Transmission will be ended.	The process of closing the communication port has timed out.	Verify that the instrument is switched ON, and that the instrument connection and communication port settings are correct.
Instrument initialization has timed out.	The process of initializing the instrument has timed out.	Determine if there is a problem with the instrument operation, or if there is a problem with communication between the PC and the instrument.
The selected communication port is already open. Verify that it is not used with another connection.	An attempt was made to open a communication port that has already been opened.	Determine if the selected port is being used with another connection.
Cannot open communication port. Check instrument status and transmission settings.	The communication port failed to open.	Verify that the instrument is switched on, and that the instrument connection and communication port settings are correct.
Mode error: Check instrument condition. If necessary, restart instrument.	The operation was not accepted due to the instrument condition.	Verify the instrument operation. If necessary, restart the instrument.

Table 5.3• Error Message List 3

Error Message	Description	Corrective Action
ERROR: Syringe Zero Point Detection.	Syringe zero point detection could not be performed. Syringe is not properly installed.	Properly mount the syringe. Then select Zero Point Detection from the Maintenance menu.
ERROR: Vial rack not present.	Operation cannot continue because there is no ASI vial rack in the turntable.	Install a vial rack, and then execute the operation again.
ERROR: Vial rack has been exchanged.	Measurement cannot be performed because the ASI vial rack specified in the settings differs from the type actually detected.	Verify the type of vial rack installed in the ASI-V, and then set the vial rack type in the software.
ERROR: Vial rack type not set.	The measurement parameters cannot be validated because the ASI vial rack type is not specified in the software.	Set the vial rack type in the software.
ERROR: ASI Cover is open.	The ASI-V cover is open. The ASI-V operation has stopped as a safety measure.	Close the ASI-V cover.

The following messages indicate that an error has occurred that prevents measurement from continuing. Take the appropriate corrective actions as indicated in the following table. If the cause of the problem cannot be discovered, or if the same error message appears after taking the indicated corrective actions, contact your Shimadzu representative.

Table 5.4• Error Message List 4

Error Message	Description	Corrective Action
ERROR: ROM Write Error	An error occurred while writing to memory.	Switch Off the power to the instrument. Contact your Shimadzu representative.
ERROR: Syringe Position Detection	The syringe is not operating normally.	Switch Off the power to the instrument. After switching On the power and running the instrument again, see if the same error is generated.
ERROR: Syringe Operation	The syringe is not operating normally due to clogging of the tubing connected to the 8-port valve.	Switch Off the instrument, and remove any foreign particles from the line. After switching on the power and running the instrument again, see if the same error is generated.
ERROR: Syringe Zero Point Detection	The syringe is not operating normally.	Switch Off the instrument. After switching On the power and running the instrument again, see if the same error is generated.
ERROR: 8-Port Valve Position Detection	The 8-port valve is not operating normally due to clogging inside of the sample injector 8-port valve.	Switch Off the instrument, and remove the rotor from the 8-port valve. Remove any particles found in the 8-port valve and wash valve.
ERROR: ASI Arm Vertical Position Detection	The ASI-V arm is not operating normally in the vertical direction.	Switch Off the power to the TOC-V main instrument. After switching on the power and running the instrument again, see if the same error is generated. Since the problem is unrelated to the TOC-V main unit, disconnect the ASI-V from the main unit, and perform operations using the TOC-V main unit alone until normal operation of the ASI-V is restored.
ERROR: ASI Arm Vertical Movement	The ASI-V arm is not operating normally in the vertical direction.	
ERROR: ASI Arm Horizontal Position Detection	The ASI-V arm is not operating normally in the horizontal direction.	
ERROR: ASI Arm Horizontal Movement	The ASI-V arm is not operating normally in the horizontal direction.	
ERROR: ASI Turntable Position Detection	The ASI-V turntable position could not be detected. Either the rack is not properly seated in the turntable, or some object may be blocking a position detection notch on the periphery of the rack.	Switch Off the power to the TOC-V main unit. Make sure the rack is properly seated, and remove any objects that may be blocking the position detection notch on the periphery of the rack. After eliminating the cause of the problem, place the cover on the ASI-V, and switch on the power to the instrument again.
8-Port Sampler 1 position detection	8-Port Sampler 1 operation error occurred due to foreign matter infiltration into 8-port valve, etc.	Switch Off the instrument. Remove 8-port valve rotor from 8-Port Sampler 1, remove debris, etc., and wash valve.
8-Port Sampler 1 operation		
8-Port Sampler 2 position detection	8-Port Sampler 2 operation error occurred due to foreign matter infiltration into 8-port valve, etc.	Switch off instrument. Remove 8-port valve rotor from 8-Port Sampler 2, remove debris, etc., and wash valve.
8-Port Sampler 1 position detection	8-Port Sampler 1 operation error occurred due to foreign matter infiltration into 8-port valve, etc.	Switch off instrument. Remove 8-port valve rotor from 8-Port Sampler 1, remove debris, etc., and wash valve.

5.6.2 Troubleshooting

5.6.2.1 TOC-V

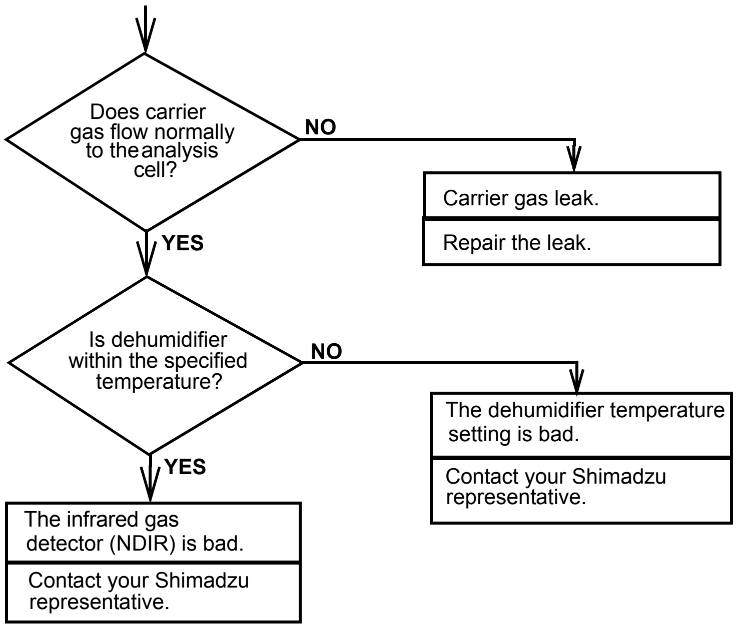
1) An error message is displayed:

   Refer to the error message.

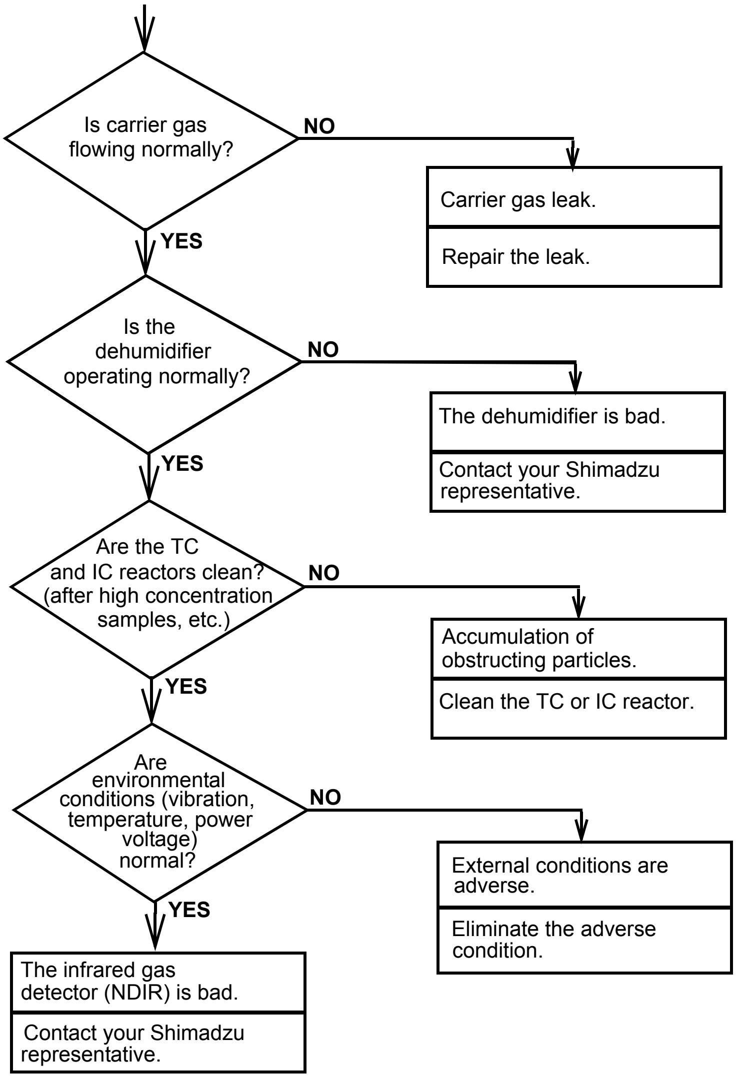
2) The Ready lamp will not light:

   Refer to the “Monitor” screen to see which parameters are not satisfied.

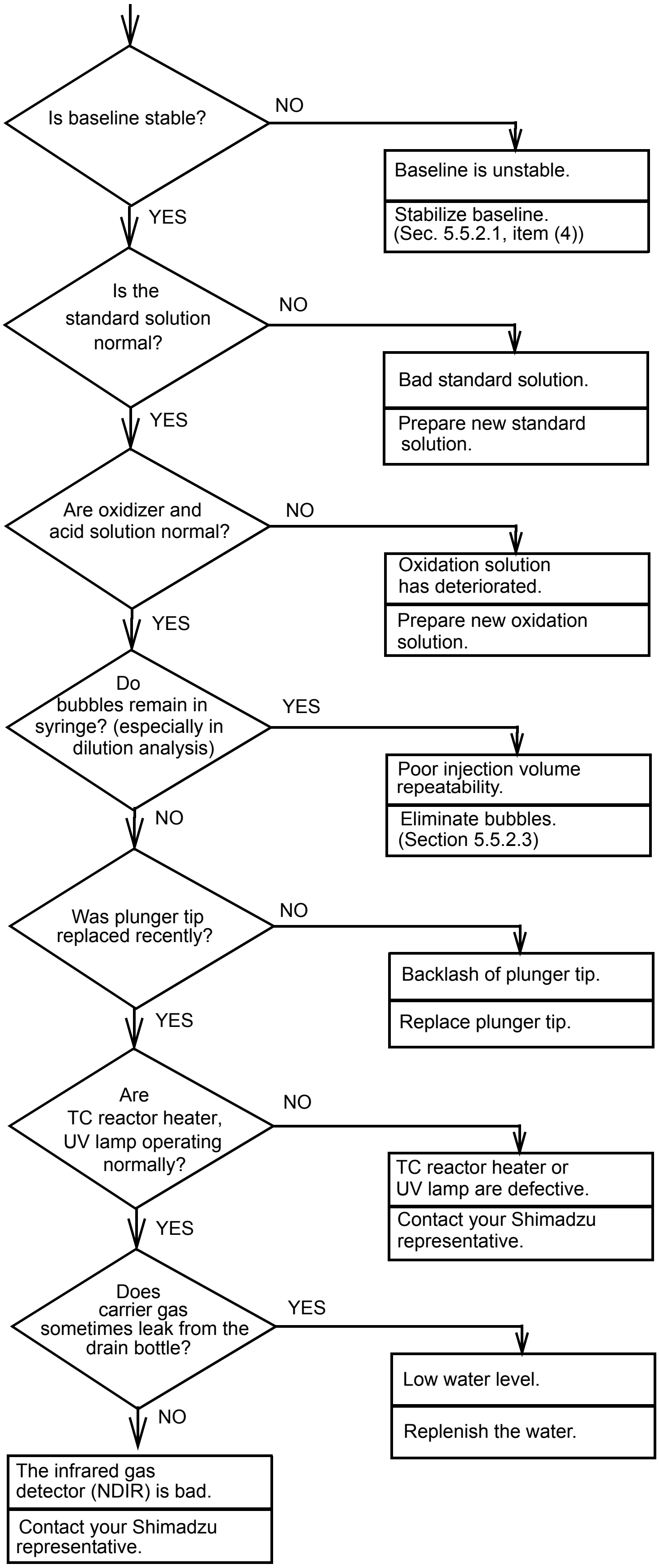
3) The baseline stays over the auto zero range. Baseline position never reaches the ready state.



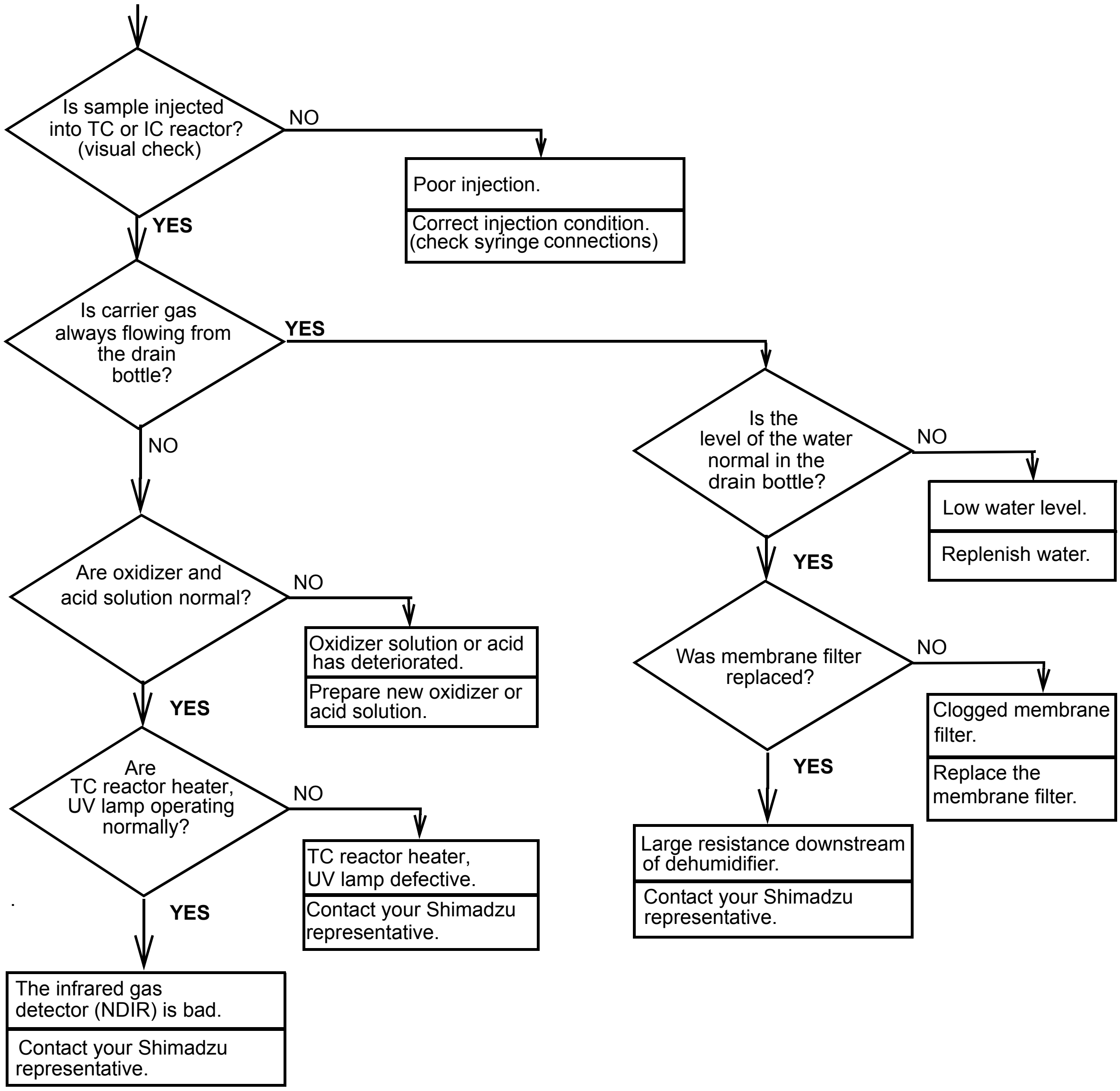
4) The baseline is unstable.



5) Poor reproducibility with a standard solution.

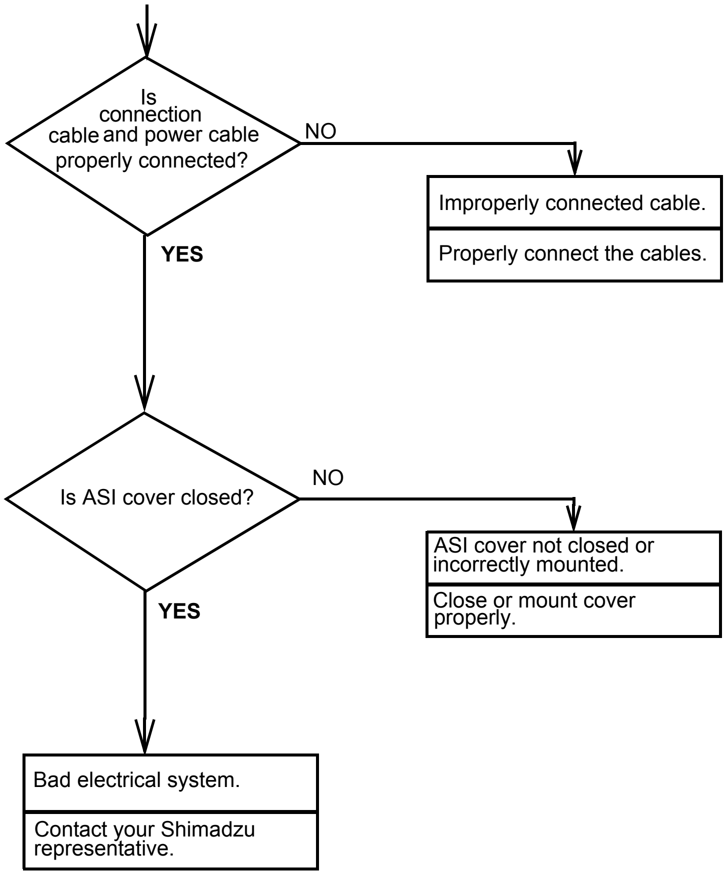


6) No peaks appear after sample injection.

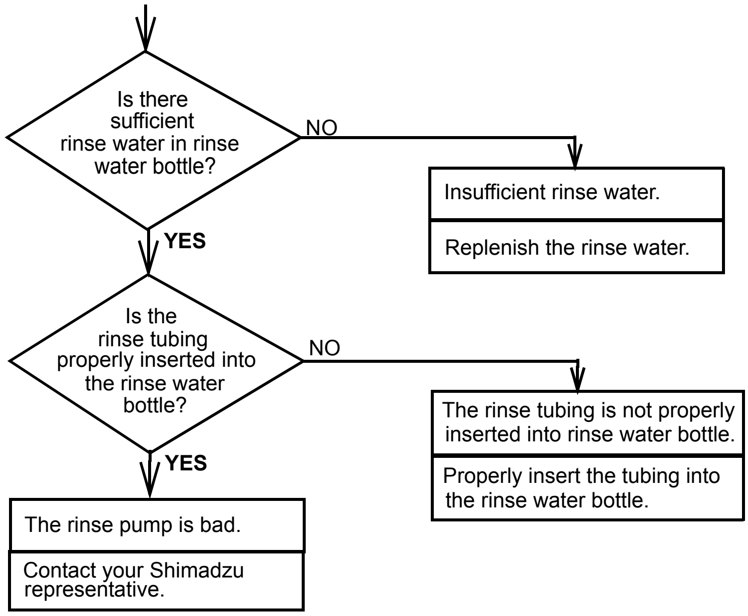


5.6.2.2 ASI-V

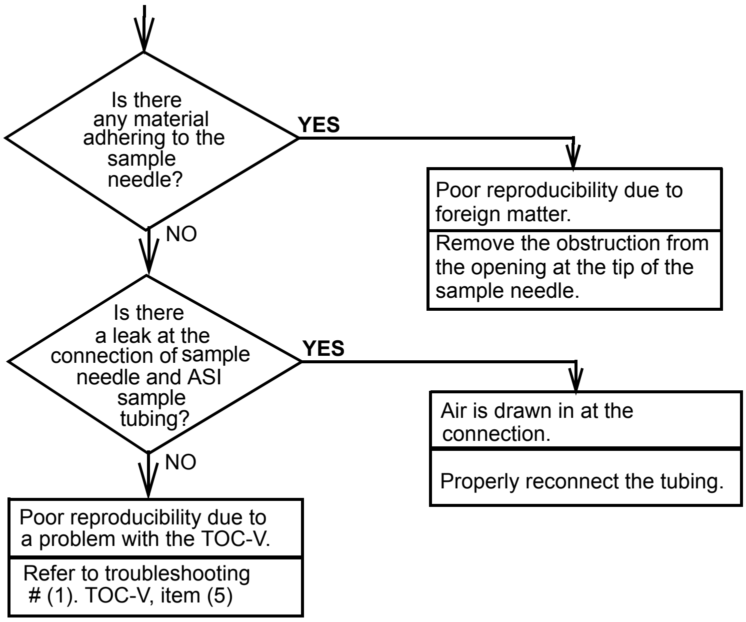
1) The ASI-V does not operate.



2) Rinse water is not delivered from the rinse port.

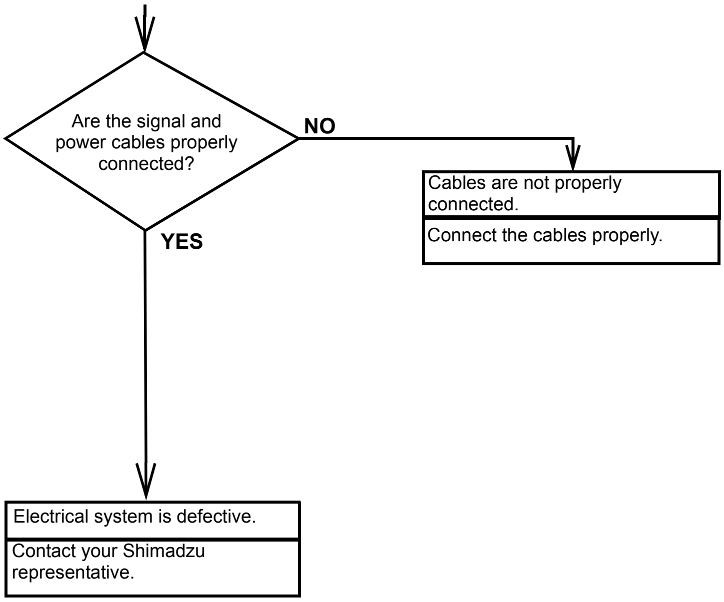


3) Poor measurement reproducibility.

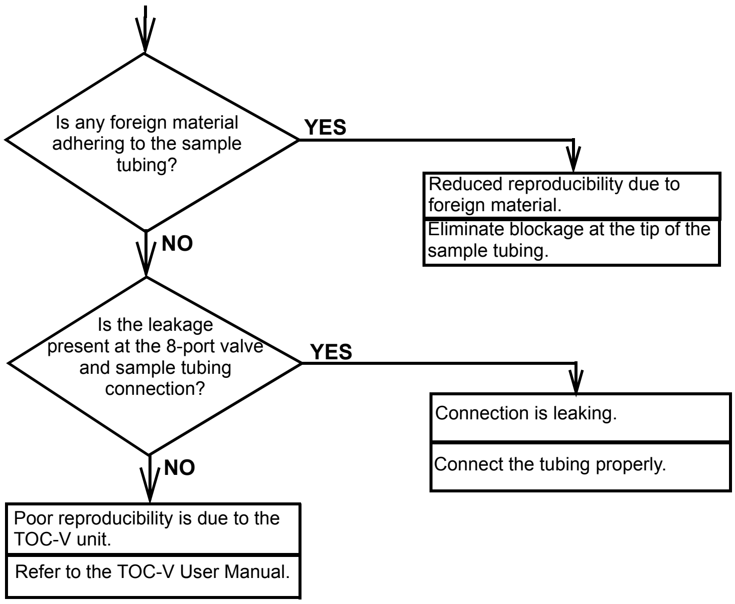


5.6.2.3 8-Port Sampler OCT-1

1) 8-Port Sampler does not operate.



2) Poor reproducibility.



5.6.2.4 Corrective Actions for Poor Reproducibility

Corrective Action to Eliminate Bubbles Inside the Syringe

Small bubbles that adhere to the inside of the syringe by the plunger tip have little effect on analysis values. Larger bubble inside the syringe, however, will affect values and result in poor reproducibility. Large bubbles form easily inside of a dirty syringe. These bubbles are evident when sample is mixed with dilution water in the syringe and when sparging inside the syringe for IC removal pretreatment for NPOC analysis.

To clean the syringe, detach the syringe from the syringe pump. Repeatedly fill the syringe with laboratory glassware detergent, discharging the cleaning solution each time. Draw a full stroke of cleaning solution into the syringe barrel and allow it to set for at least 20 minutes to overnight for heavily soiled syringes. Thoroughly rinse away all of the detergent and reattach the syringe to the syringe pump.

Decreased Reproducibility Due to Plunger Tip Backlash

With use, the plunger tip wears in the following ways.

1) The surface of plunger tip becomes worn, and the seal between plunger tip and the inside of the glass syringe barrel deteriorates, allowing slight amounts of water to leak out with vertical movements of the plunger.

2) The contact site of the plunger tip and plunger becomes loose, causing a slight backlash with vertical movement of the plunger.

When the above conditions start to occur, repeatability of sample injection volume decreases, resulting in diminished analysis reproducibility. Item (2) above, rapidly leads to decreased reproducibility. It is very difficult to determine backlash by inspecting movement of the plunger tip, Inspect for backlash as follows.

Before sample injection, observe the wash injection used to replace the previous sample in the syringe and flow line with the new sample. After injection, if a droplet forms at the tip of the injection tubing and increases in size, there is a strong possibility that backlash has increased. The corrective action is to replace the plunger tip.

Note: The effect of backlash increases with smaller sample injection volumes. Poor results might be obtained with a sample injection volume of 10µL, while the effect might be negligible with a 100µL sample injection volume.

6

Reference Materials

This chapter describes the principles of analysis and lists instrument specifications, accessories, and parts. This chapter also includes installation procedures, for use in the event that the instrument is relocated.

6.1 Principles of Analysis

There are two types of carbon present in water: organic carbon and inorganic carbon. Organic carbon (TOC) bonds with hydrogen or oxygen to form organic compounds. Inorganic carbon (IC or TIC) is the structural basis for inorganic compounds such as gas carbonates and carbonate ions. Collectively these are referred to as total carbon (TC) and have the relationship TOC=TC-IC.

Following is a description of the TOC measurement principle.

6.1.1 Principle of TC (Total Carbon) Analysis

In the TOC-V, carrier gas flows at a controlled rate of 200mL/min through the TC reactor, which is comprised of a UV lamp and heater. When sample is injected along with the oxidizing reagent (containing sodium persulfate and phosphoric acid) into the TC reactor which has been heated to 80°C, the TC in the sample is oxidized and decomposed to form carbon dioxide. This carbon dioxide is swept via the carrier gas from the reaction tube to a dehumidifier for cooling and dehydration. These products then pass through a halogen scrubber to reach the cell of a non-dispersive infrared detector (NDIR), where the carbon dioxide is detected. The analog detection signal of the NDIR forms a peak, and the area of this peak is measured by a data processor.

The peak area is proportional to the TC concentration of the sample. Therefore, when a TC standard solution has been analyzed to create a calibration curve equation expressing the relationship between TC concentration and peak area, the TC concentration in the sample can be calculated.

6.1.2 Principles of IC (Inorganic Carbon) Analysis

The IC comprises the carbon within carbonates, hydrogen carbonates and dissolved carbon dioxide.

Defining IC

The IC (inorganic carbon) in TOC analysis refers to carbon contained in the carbon dioxide dissolved in water and that found in carbonates. By acidifying the sample with a small amount of phosphoric acid to obtain a pH less than 3, all the carbonates produce carbon dioxide (CO_2) according to the following reaction.

$$Me_2CO_3 + H_3PO_4 \rightarrow CO_{2(g)} + 2MeCl + H_2O$$

$$MeHCO_3 + H_3PO_4 \rightarrow CO_{2(g)} + MeCl + H_2O$$

The carbon dioxide and dissolved carbon dioxide in the sample are volatilized by bubbling (sparging) gas through the sample.

Analysis with the IC Reactor

Sparge gas flows at a controlled rate of 200mL/min through the IC reactor. When sample is injected along with phosphoric acid into the IC reactor, only the IC component of the sample is converted to carbon dioxide, which is subsequently detected by the NDIR. The IC concentration in the sample is then measured in the same way as the TC concentration.

6.1.3 Principle of NPOC (Non-Purgeable Organic Carbon) Analysis

After acidifying the sample to pH 2 to 3 by adding phosphoric acid, sparge gas is bubbled through the sample to eliminate the IC component. The TC remaining in the sample after sparging is measured to determine total organic carbon, and the result is generally referred to as (acidify and sparge) TOC. However, in the TOC-V, this analysis value is referred to NPOC to distinguish it from the TOC value obtained by the difference between TC and IC. NPOC stands for non-purgeable organic carbon and refers to the non-volatile organic carbon contained in a sample.

NPOC and TOC (obtained by IC elimination) described in the TOC-related standard methods and referred to in water quality-related test methods (JIS, ASTM, EPA, EN) are identical. If the sample contains purgeable organic substances, this component can be lost during the sparging process. Consequently, when the sample contains purgeable organic substances, TOC should not be measured by the NPOC method. If the dissolved purgeable organic component in the water sample is large, the amount volatilized during sparging is relatively small. Generally, the amount of purgeable organic substances in natural environmental water, public water and purified water is small, so NPOC is referred to as TOC.

Table 6.1 Residual Rate of Volatile Organic Substances Using a Nitrogen Gas Sparge

Organic Substance	Pre-Sparging Conc. (ppm)	Post-Sparging Conc. (ppm)	Residual Rate (%)
Methanol	117. 5	116	98.6
Ethanol	106.5	105	98.5
Isopropyl alcohol	129	127	98.5
n-butyl alcohol	117	115	98.3
Acetone	106	101	95.3
Acetoaldehyde	130	117	90.0
Ethyl acetate	102	88	86.3
Tyrosine	117	116.5	99.5
Benzene	85	2.5	2.9
Cyclohexane	79	2	2.5

6.1.4 Principles of Measuring TOC

The TOC-V employs 2 methods for TOC analysis.

- TC-IC Method
- NPOC Method

TC-IC Method

The TOC value determined by the difference in TC and IC analysis values includes errors associated with both the TC and IC analyses. This results in a large error for the TOC value. NPOC analysis is generally used and is recommended for samples that contain more IC than TOC (samples where TC consists almost entirely of IC).

NPOC Method

This is the most widely used method of TOC analysis. There is a tendency for samples that become foamy during sparging to partially form bubbles and flow out of the syringe, along with the concentrated TOC content of the sample, thus leaving a smaller concentration of TOC in the syringe. The TC-IC method is recommend for samples that foam during the sparging process.

6.2 Analysis-Related Technical Information

6.2.1 Peak Area Analysis

When a sample is introduced, the TOC-V automatically detects the beginning and end of each peak produced and calculates the peak areas. Detection of the beginning and end of each peak is based on the slope of the tangent, which changes moment to moment. Peak detection begins when that slope exceeds a predetermined value and ends when a negative slope falls below a specified value.

Note: Baseline correction is performed for peaks produced with fluctuating baseline to obtain the correct peak area.

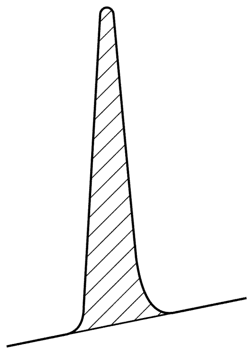


Figure 5.1 Determining Peak Area

6.2.2 Calibration Curves

6.2.2.1 Types of Calibration Curves

1-Point and 2-Point Calibration Curves

The output signals of the TOC-V are linearized. There are almost no factors in the reaction systems of the TC and IC reactor components that might cause the concentration-output characteristic to deviate from linearity. Consequently, calibration curves are normally generated as 1-point curves consisting of 1 span point, or 2-point curves consisting of 1 span point and the zero point.

The concentration of TC and IC vary with standard solutions prepared with purified water. A calibration curve generated using concentrations greater than 50 - 100mg/L will pass very close to the zero point. In this case, it is practical to generate a 1-point calibration curve.

Use a 2-point calibration curve when the concentration of TC or IC in the purified water used to prepare the standard solutions cannot be disregarded with respect to the standard solution concentration. Refer to Section 6.2.2.2 “Shifting of Calibration Curves” for information on handling of this type of curve.

Multiple Point Calibration Curves

Calibration curves can be generated using up to 10 points. Calibration curves consisting of 3 or more points can be generated as a point-to-point curve or as a regression line using the least squares method. A correlation coefficient is displayed with the least squares regression method.

6.2.2.2 Shifting of Calibration Curves

Shift to Origin

This function is used when the amount of TC or IC in the purified water used for standard solution preparation cannot be ignored with respect to the standard solution concentration. A correction must be made by shifting the calibration curve in a parallel fashion so that it passes through the origin. This accounts for the TC or IC concentration in the water used for standard solution preparation.

When water containing 0.5mg/L TC is used to prepare a 10mg/L TC standard solution (actually 10 + 0.5mg/L TC), a 2-point calibration curve appears as shown by the solid line in Figure 6.2 “Shifting the Calibration Curve”. If this curve is used as is, the sample analysis values will always show a value 0.5mg/L less than the true value. By shifting the calibration curve so that it passes through the origin, as shown by the broken line in “Figure 6.2”, the calibration curve deviation is effectively corrected.

When there is a system blank, the measured value is greater by the amount of the system blank. The system blank refers to the peak obtained by measuring water that contains absolutely no carbon (water with zero TC). This instrument allows shifting of calibration curves only to the extent of the system blank value. In most cases, analysis error due to the TC content in the water used in preparing standard solutions is much greater than the system blank value. Correcting the calibration curve based on the TC content in the standard solution preparation water will produce satisfactory analyses. By this method, the analysis value obtained for a system blank, would be greater than zero by the amount of this blank value.

Shift to Blank Point

This shift procedure is performed in high sensitivity analysis where the system blank value could have an effect on the analysis value.

By selecting the shift-to-blank point option, a TC blank check analysis is performed using the same injection volume that is used in the analysis. The calibration curve zero point is shifted to the TC blank check value.

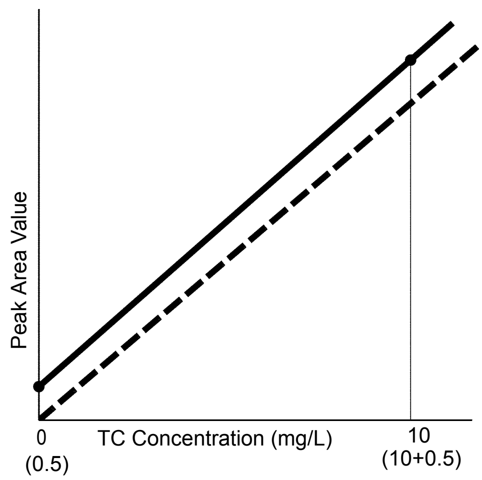


Figure 6.2 Shifting the Calibration Curve

6.2.3 Sparging During Standard Solution Analysis

For standard solutions that have a TC concentration so low as to be affected by the amount of dissolved carbon dioxide in the standard solution, perform analysis of the standard solution after it has been sparged with carrier gas. The dissolved carbon dioxide is eliminated by sparging and will no longer affect the concentration of the standard solution.

The amount of dissolved carbon dioxide in the water used for standard solution preparation is affected by factors such as water purification method, length and condition of storage, and the concentration of carbon dioxide in the atmosphere. Even if the dissolved carbon dioxide concentration in the water is low, the process of preparing the standard solution it may expose it to the atmosphere. Refer to Table 6.2 "CO_2 Content (ppm) in Distilled Water Equilibrated with Atmosphere Temperature (°C)" which presents the dissolution rates of atmospheric carbon dioxide in water.

Both the concentration of the standard solution and the concentration of TOC contained in the water must be taken into consideration when deciding whether to use the sparging function. Use of sparging is indicated if the TOC contained in the water is low enough to not substantially affect the standard solution concentration, but not low enough to be ignored.

Note: Do not use sparge IC standard solutions as sparging decreases the IC concentration.

Table 6.2 CO_2 Content (ppm) in Distilled Water Equilibrated with Atmosphere Temperature (°C)

Atmospheric CO_2 (vol%)	0	5	10	15	20	25	30
0.030	1.00	0.83	0.79	0.59	0.51	0.44	0.38
0.033	1.10	0.91	0.76	0.65	0.56	0.48	0.42
0.044	1.47	1.22	1.02	0.87	0.74	0.64	0.56

The above CO_2 content can be converted to IC using the following equation.

$$\text{IC (ppm)} = CO_2\ \text{(mg/L)} \times 0.27$$

6.2.4 Automatic Selection of the Optimal Calibration Curve

The TOC-V has the ability to automatically select which calibration curve is used to determine the sample value. Up to 3 calibration curves can be specified in the "Measurement Parameters" screen. If more than one calibration curve is listed, the TOC-V uses the following scenarios to determine the optimum calibration curve for analysis.

Procedure

Analysis is performed using the calibration curve designated as Calib. (1st).

Selection Result

1) If the sample value is less than the concentration of Calib. (1st), and greater than the conc of Calib (2nd), the first calibration curve is used:

```
Conc. of 1st Calibration Curve > Measured Value > Conc. of 2nd
Calibration Curves → First calibration curve is used.
```

2) When the concentration of Calib (2nd) is less than Calib. (1st), a sample value that is less than both is calculated using the lower concentration of Calib. (2nd):

```
Conc. of 1st Calibration Curve > Conc. of 2nd Calibration Curve >
Measured Value → 2nd Calibration Curve is used.
```

3) When three calibration curves are specified in the "Measurement Parameters" screen, the calibration curve that is closest to the sample value without being less is the one used for calculation of the result.

```
Conc. of 1st Calibration Curve > Conc. of 2nd Calibration Curve >
Measured Value > Conc. of 3rd Calibration Curve → 2nd
Calibration Curve is used.
```

```
Conc. of 1st Calibration Curve > Conc. of 2nd Calibration Curve >
Conc. of 3rd Calibration Curve > Measured Value → 3rd
Calibration Curve is used.
```

4) If the sample value is greater than the concentration of Calib. (1st), and the concentration of Calib. (2nd or 3rd) is greater than the concentration of Calib. (1st):

```
Conc. of 2nd or 3rd Calibration Curve > Measured Value > Conc. of
1st Calibration Curve →2nd or 3rd Calibration Curve is used.
```

5) If the measured value is greater than the concentration of the first calibration curve and the concentrations of the other two calibration curves (2nd or 3rd) are greater than the concentration of the first calibration curve:

   Analysis is first performed using the calibration curve with the lower concentration of the other two.

```
Measured Value > Conc. of 2nd or 3rd Calibration Curve > Conc. of
1st Calibration Curve →2nd or 3rd Calibration Curve is used.
```

Note: By setting the concentration ratio between calibration curves to a factor of approximately 10, this function can be effectively utilized to enable analyses covering a wide range of concentrations.
Example:

`Calib. (1st): 10ppm, Calib. (2nd): 100ppm, Calib. (3rd): 1000ppm`

Considerations for Auto-select Function

- The above processing does not change even if multiple point calibration curves are included.
- When the number of analyses is set to more than one, the above determination is made using the measured value of the first analysis.
- Since analysis always begins from the first calibration curve, set the most-used calibration curve in this position.
- The priority of the second and third calibration curves is determined on the basis of the calibration curve concentrations and the sample concentration.

6.3 Specifications

6.3.1 TOC-VWP

Analyte	TC, IC, TOC (TC-IC), NPOC
Measurement Principle	Wet chemical (Heated - UV - Persulfate) oxidation with non-dispersive infrared detection (NDIR)
Measuring range	0 - 35000mg/L
Detection limits	0.5µg/L
Measurement time	TC and IC: Approx. 4min NPOC: Approx. 5 min.
Repeatability	CV% within 1.5% (CV% within 2.0% if range exceeds 100mg/L)
Sample introduction	Auto injection using an 8-port valve syringe pump
Sample injection volume	350 - 20400µL (variable)
Sample dilution function	Dilution within syringe, dilution factor 2 - 50 times
Pretreatment for IC	Automatic acid addition and sparging
Carrier gas	High purity nitrogen Pressure: Between 300-600kPa (44-87PSI)
Carrier gas flow rate	200mL/min (400mL/min when performing sparging for NPOC)
Ambient temperature	5 - 35°C
Power requirements	AC100-127V (for a 100V system) or AC220-240V (for a 200V system)
Fuse	4A, T
Approximate dimensions	(W) 440mm x (D) 560mm x (H) 460mm (excluding projections)
Weight	Approximately 40kg

6.3.2 ASI-V

Sample containers	24mL vial rack: 24mL (External diameter 23mm x Height 85mm), 100 vials, 100 septum-equipped caps
	40mL vial rack: 40mL (External diameter 27mm x Height 95mm), 72 vials, 72 septum-equipped caps
	125mL vial rack:125mL (External diameter 48mm x Height 112mm), 24 vials, 24 septum-equipped caps
Vial rack capacity	24mL vial rack: 93 vials
	40mL vial rack: 68 vials
	125mL vial rack: 24 vials
Sample pretreatment	Acid addition and sparging possible for NPOC analysis
	Needle rinse possible
Ambient temperature	5 - 35°C
Approximate dimensions	(W) 370mm x (D) 540mm x (H) 490mm (excluding projections)
Weight	Approximately 13kg

6.3.3 OCT-1 8-Port Sampler

No. of sample containers accommodated	8 containers
	16 containers (using 2 OCT-1 units)
Ambient temperature	5 - 35° C
External dimensions	Approx. (W) 245 x (D) 245 x (H) 440mm (excluding protruding parts)
Weight	Approx. 3.5 kg

6.3.4 PC Hardware Requirements

PC	IBM-PC/AT or compatible, with 9-pin RS-232C connector
CPU	233MHz or better
Memory	32 MB or greater
Hard drive	2 GB or greater
OS	Windows95, Windows98, Windows2000, WindowsNT 4.0 (SP3 or later), WindowsXP (*For compliance with FDA 21 CFR Part 11, use Windows2000) The User Authentication function cannot be used with WindowsXP.
Monitor resolution	800 x 600 or better, 1024 x 768 suggested

6.4 Standard Accessories

Table 6.3 Standard Accessories List 1 (TOC-VWP)

Part Number	Part Name	Quantity
638-52602	TC reactor	1
638-52592	IC reactor	1
631-81280	TC reactor heat transmitting sheet	1
638-52336-06	Persulfate bottle 500mL	1
638-52336-04	Acid bottle 500mL	1
638-52337	Rinse water bottle	1
630-00999	CO_2 absorber	1
631-81214	CO_2 absorber platform	1
638-59214	25mL syringe	1
638-59214-01	Plunger tip	1
630-00635-01	Potassium biphthalate, 25g	1
630-00962-01	Sodium carbonate, 25g	1
630-00963-01	Sodium bicarbonate, 25g	1
630-00315-09	Plastic tube 9mm ID x 12mm OD	2m
071-60813	Adapter, KPR-18	1
071-60814-01	Power cord set	1
072-02004-19	Fuse, 218 002	2
631-20265	Plug, 35154	1
631-78002	Wrench, for 8-port valve	1
037-72138-30	Caution label (corrosion caution), for drain bottle	1
072-60359-01	Wiring band	3
638-64727	CD-ROM “TOC-Control V”	1
638-74029	Cable (RS-232C)	1
638-94247	English Instruction Manual	1

Table 6.4 Standard Accessories List 2 (ASI Unit)

Part Number	Part Name	40mL	125mL
038-00165-21	Vials, 40mL (with cap, septum)	72	
038-00165-32	Vials, 125		24
638-20074-02	Cap, 125mL		24
038-00165-49	Septum, 125mL		100
638-52337	Rinse water bottle	1	1
072-60310-03	Wiring band, TM-53M	2	2
631-51869-20	Name plate, SAMPLE	1	1
631-51869-21	Name plate, GAS	1	1
072-60301	Wiring band, Nylon TY-23M	1	1
638-74027	Cable ASSY, ASI, signal	1	1
638-41448-01	Needle, Standard Type 24, 40	1	
638-41449-02	Needle, Standard Type 125		1

Table 6.5 Standard Accessories List 3 (OCT-1 8-Port Sampler)

Part Number	Part Name	8-Port Sampler 1	8-Port Sampler 2	Details
631-20265	8-port valve rotor	1	1	Rotor (for replacement maintenance)
638-41481-02	Flared tubing set W	1*		8 sample tubes,1 tube for connection to main unit.**
638-41481-04	Flared tubing set W2		1*	8 sample tubes,1 tube for connection to main unit.***
638-74004-01	Power cable	1		Main unit connection cable.
638-74028-01	Signal cable	1		Main unit connection cable.
638-74028-02	Power/signal cable		1	Connection cable for 8-Port Sampler 1, 8-Port Sampler 2

* Flared tubing set W and W2 are for samples and for connecting the main unit. Both have 1.5 mm internal diameter. They are supplied with wet type 8-port sampler.
** There is a No. 2 mark at the TOC-V main unit. Connect between the port No. 2 of the TOC-V main unit 8-port valve and the COM port of the 8-port valve of the 8-Port Sampler 1.
*** There is a No. 1 mark at the TOC-V main unit. Connect between the port No. 1 of the TOC-V main unit 8-port valve and the COM port of the 8-port valve of the 8-Port Sampler 2.

6.5 Special Accessories

Table 6.6 Special Accessories List 1

Part Number	Part Name	Comment
638-93141-10	Autosampler ASI-V (40mL)	40mL vials
638-93141-06	Autosampler ASI-V (125mL)	125mL vials
638-53044-02	40mL vial rack set for autosampler	Set consists of 40mL vial rack + 40mL vial caps + septa
638-53044-03	125mL vial rack set for autosampler	Set consists of 125mL vial rack + 125mL vial caps + septa
638-93150-04	OCT-1 8-Port Sampler	For TOC-VWP
638-93150-12	OCT-1 8-Port Sampler (2nd unit)	For TOC-VWP
638-77152	External sparge kit	
630-00749-04	High purity nitrogen gas cylinder	Recommended that this be obtained locally.
630-08585-05	Cylinder pressure regulator	User can provide this item.
638-41204	Air supply tubing set (with 20 meters of tubing)	User can provide this item.
630-02525-01	High speed homogenizer	
618-52344	Drain Bottle Kit for autosampler	

Table 6.7 Special Accessories List 2 (Reagents)

Part Number	Description	Quantity	
		Sodium Hydrogen Persulfate (500g)	Phosphoric Acid (500mL)
638-60149-02	TOC-VWP Reagent Kit (single use supply)	1	1
638-60149-03	TOC-VWP Reagent Kit (1-year supply)	7	3

6.6 Consumable Parts List

Table 6.8 Consumable Parts List 1 (TOC-VWP)

Part Number	Part Name	Comment
630-00999	CO_2 absorber	
630-00992	Halogen scrubber	
630-00635-01	Potassium biphthalate, 25g	
630-00962-01	Sodium carbonate, 25g	
630-00963-01	Sodium bicarbonate, 25g	
631-20265	8-port valve rotor	
638-69134	UV lamp	
638-59214-01	Plunger tip	
638-92207-01	Consumable Parts Kit (TOC-VWP)	Refer to "Table 6.11" for contents.
638-60149-02	TOC VWP Reagents Kit (single use supply)	Refer to "Table 6.12" for contents.
638-60149-03	TOC-VWP Reagents Kit (1 year supply)	Refer to "Table 6.12" for contents.

Table 6.9 Consumable Parts List 2 (ASI-V)

Part Number	Part Name	Comment
038-00165-21	Vial set *	40mL, 72pcs.
038-00165-32	Vials	125mL, 24pcs.
038-00165-50	Septum	40mL, 100pcs.
038-00165-49	Septum	125mL, 100pcs.
038-00165-40	Cap	40mL, 200pcs.
638-20074-02	Cap	125mL, 24pcs.
042-00405-11	Rinse pump head	

* Each vial set contains septa and caps in the same quantity as the vials.

Table 6.10 Consumable Parts List 3 (8-Port Sampler OCT-1)

Part Number	Part Name	Comment
631-20265	8-Port valve rotor	

Table 6.11 Consumable Parts List 4 (TOC-VWP)

Part Number	Part Name	Comment
630-00999	CO_2 absorber	1
630-00992	Halogen scrubber	2
630-00635-01	Potassium biphthalate, 25 g	1
630-00962-01	Sodium carbonate, 25 g	1
630-00963-01	Sodium bicarbonate, 25 g	1
638-69134	UV lamp	1
631-20265	8-port valve rotor	1
638-59214-01	Plunger tip	2

Table 6.12 Consumable Parts List 5 (Reagents)

Part Number	Description	Quantity	
		Sodium Hydrogen Persulfate (500g)	Phosphoric Acid (500mL)
638-60149-02	TOC-VWP Reagent Kit (single use supply)	1	1
638-60149-03	TOC-VWP Reagent Kit (1-year supply)	7	3

6.7 Maintenance Parts List

Table 6.13 Maintenance Parts List 1 (TOC-VWP)

Part Number	Part Name	Comment
631-81280	TC reactor heat transmitting sheet	
631-52262	TC reactor tube	TC reactor glass tube part only (excludes UV lamp)
638-42030	IC reactor tube	IC reactor glass tube part only
638-56135	8-port valve	
638-59214	Syringe	
631-41660	Flared tubing	
631-41660-05	Flared tubing, for dilution water	
046-00044-11	Membrane filter	
638-65383	Tandem cell (in case)	

Table 6.14 Maintenance Parts List 2 (ASI-V)

Part Number	Part Name	Comment
638-41450-01	Needle, Wet Type 40mL	For 40mL, 1pc.
638-41450-02	Needle, Wet Type 125mL	For 125mL, 1pc.
638-41448-01	Needle, sparging	For 40 mL sparging, 1pc.
638-41449-01	Needle, simultaneous sparging	For 40 mL simultaneous sparging, 1 pc.
638-42029-04	Tubing set (sampling)	For 40/125mL, 1 sampling tube, w/tube retainer, tube seal
638-42029-05	Tubing set (sparge)	1 sparge tube, w/ tube retainer, tube seal
638-42029-03	Tubing set (simultaneous sparge, sampling and sparge)	1 each simultaneous sparge and sampling tube, w/ tube retainer, tube seal

Table 6.15 Maintenance Parts List 3 (OCT-1 8-Port Sampler)

Part Number	Part Name	Comment
631-20265	8-port valve rotor	Spare rotor
638-41660	Flared tubing C	Sampling tube; for wet type TOC-V
638-41443-05	Common tubing C	Main unit - OCT-1 connection tube; for wet type TOC-V, 1 unit
638-41443-06	Common tubing C2	Main unit - OCT-1 connection tube; for wet type TOC-V, add-on

6.8 Installation

6.8.1 Before Installation

Parts Inspection

Confirm that all of the components listed in Section 6.4 "Standard Accessories" are included in the shipping package.

6.8.2 Installation Site

When selecting an installation site consider the size of the instrument, how it will be used, and the installation conditions indicated below. Instrument damage or analysis errors may result from improper installation.

6.8.2.1 Installation Site Selection

This instrument is designed to be placed on a workbench or similar countertop.

See Figure 6.3 "External Dimensions with ASI-V Autosampler" for a description of the external dimensions of the instrument. A space of at least 200mm is required on either side of and behind the instrument to enable unhindered operation and maintenance.

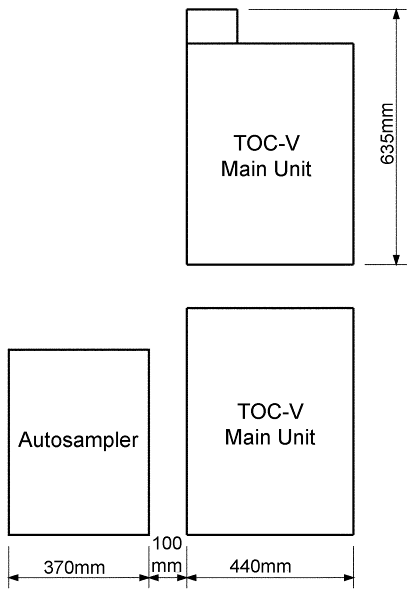


Figure 6.3 External Dimensions with ASI-V Autosampler

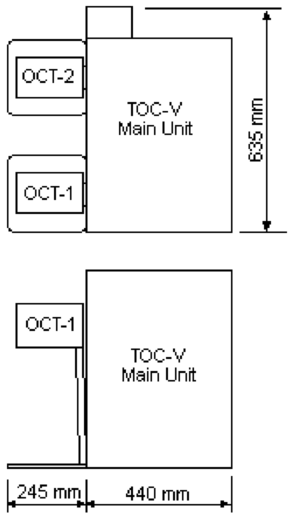


Figure 6.4 External Dimensions with OCT-1 8-Port Sampler

CAUTION

When lifting the instrument, lift from the bottom and at both sides of the instrument. Do not lift it by the front access door, as this may cause instrument damage.

6.8.2.2 Installation Site Conditions

Select an installation site that complies with the following conditions.

- A clean environment, free of corrosive gases, organic gases, and dust.

Note: Airborne contaminants will cause analysis errors in high sensitivity analyses.

- A strong, level bench, free of vibration and shock.
- A location with a stable ambient temperature.
- A location with access to a completely grounded, stable power supply.

CAUTION

Avoid locations where flames are prohibited. Some internal parts reach high temperatures and could cause a fire hazard.

Due to the possibility of analysis error, avoid the following kinds of locations.

- Locations close to heat sources or windows in direct sunlight, or in direct contact with drafts from air conditioning vents.
- Near equipment that generates intense magnetic fields, electrical fields, or high frequency waves.

6.8.3 Installation Procedure

6.8.3.1 Connecting Power Supply and Ground

Power Supply

Connect the instrument to a stable, AC100-127V (for a 100V system) or AC220-240V (for a 200V system) power supply with sufficient capacity to accommodate 6A or greater current.

CAUTION

The instrument may not operate properly if the power supply voltage exceeds the range of AC100-127V (for a 100V system) or AC220-240V (for a 200V system). Unstable voltage may cause problems during high sensitivity analysis.

Note: Since this instrument is compatible with both 50Hz and 60Hz power supplies, it can be used with either frequency.

Grounding

The included power cord is a three-conductor cable including a ground wire and a 2 prong plug (with ground) at the end. If the power outlet does not have ground receptacle, use the included 2 prong (no ground) adapter. Connect the external ground terminal to the ground.

See Figure 6.5 “Power Plug” for a description of the arrangement of the voltage terminals (AC) and ground terminal (ACC) on the power plug of this instrument. Connect them so that they match the power outlet being used.

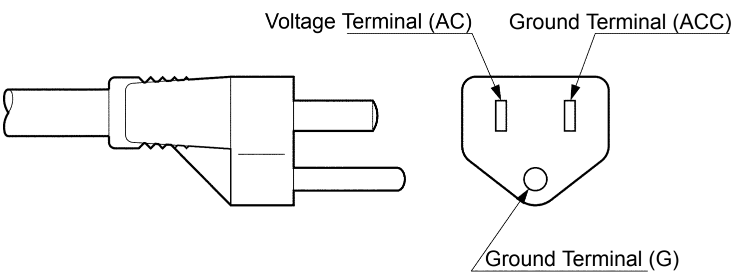


Figure 6.5 Power Plug

CAUTION

- In addition to the consideration of safety, complete grounding is necessary to prevent as much as possible the occurrence of signal noise. A Type-3 ground (ground resistance: 100W or less, see Electrical Facilities Engineering Standards for details) is recommended.
- Perform grounding separately and avoid contact with water supply pipes, gas pipes, and lightning rods.

6.8.3.2 Changing the Power Supply Voltage

The instrument was set up when it was shipped to match the power supply voltage in the locale where it will be used. If it becomes necessary to change the power supply voltage, please contact your Shimadzu representative. Depending on the voltage change, an authorized Shimadzu service engineer may be required to rewire connections to the terminal block to change the power supply voltage and/or perform changes to the main board.

6.8.3.3 Connecting Gas

Use the following types of carrier gas for this instrument.

- Cylinders filled with high purity nitrogen gas
- Instrumentation nitrogen gas

Use of Cylinders Filled with High Purity Nitrogen Gas

The gas must be guaranteed to contain less than 1 ppm of impurities consisting of carbon dioxide, carbon monoxide and hydrocarbons. Install an appropriate pressure regulator on the cylinder, and supply gas to the instrument gas connection inlet at a pressure 300kPa (43PSI).

Note: If the carrier gas contains excessive impurities, analysis accuracy is diminished and good results are not obtained.

Use of Instrumentation Nitrogen Gas

The gas must contain less than 1 ppm of impurities consisting of carbon dioxide, carbon monoxide and hydrocarbons.

Note: Install an air filter in the supply line to eliminate dust, oil mist and water droplets.

Gas Supply Pressure

Supply gas to the instrument gas connection inlet at a continuously stable pressure of 300kPa (43PSI).

When instrumentation gas is used, verify that the supplied gas pressure does not exceed 600kPa (87PSI).

⚠ Precautions for Handling Gas Cylinders

The handling and safety management of high-pressure gases is strictly regulated by high-pressure gas control laws, general high-pressure gas safety codes, and fire laws.

The gas used in this instrument is not dangerous, but mistakes in the handling of high-pressure gas cylinders can be extremely hazardous. Carefully read and obey the following precautions.

- Place gas cylinders in a well-ventilated location out of direct sunlight.
- Ensure that gas cylinders never reach temperatures higher than 40°C.
- Make sure there are no open flames anywhere within 2 meters of a gas cylinder.
- Secure cylinders with bands or other fasteners to prevent tipping and falling.
- Completely turn OFF all valves on the cylinder, immediately after use.
- Inspect the operation of the pressure gauge at least once every three (3) months.

Gas Supply Pressure

Ensure that the gas supplied to the gas inlet of the instrument maintains a constant, stable pressure of 300 - 600kPa (44 - 87PSI).

Note: Avoid a carrier gas supply pressure of greater than 600kPa (87PSI).

Gas Tubing Connections

The special accessory Air Supply Tubing Set includes nylon tubing and 2 half union connectors.

Procedure

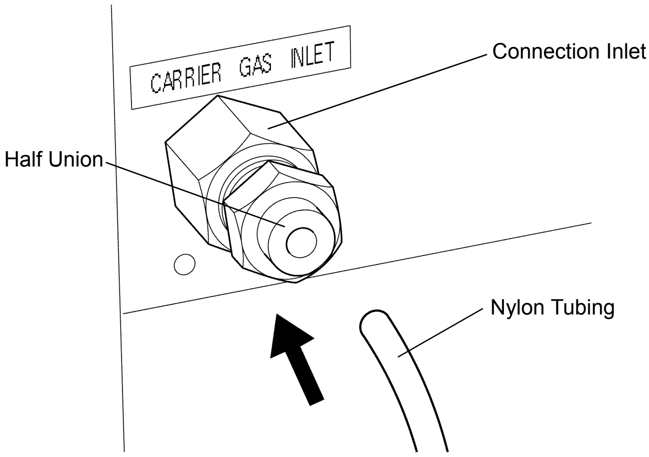


Figure 6.6 Gas Tubing Connection

1. Connect the half unions to the carrier gas inlet on the instrument and the connection outlet (¼" NPT) of the gas supply source.

2. Connect the nylon tubing (4mm OD, 2.5mm ID) between the gas supply source and the instrument.

   Note: To connect the nylon tubing, firmly insert it into the half union. To disconnect the tubing, pull the tubing off while pushing down firmly on the green-colored ring of the half union with a screwdriver.

3. Verify that there are no gas leaks.

CAUTION

- Do not allow any acute bends in the tubing.
- Clean the tubing and connectors to remove any oils or other contaminants on their inner surfaces.
- Copper tubing or stainless steel tubing may also be used.

6.8.3.4 Connecting the Drain Tubing

Drain tubing is connected to provide for external discharge of measured samples and other liquid waste. If a nearby drainage pit is not available, use a 10 to 20 liter polypropylene container as the drain receptacle.

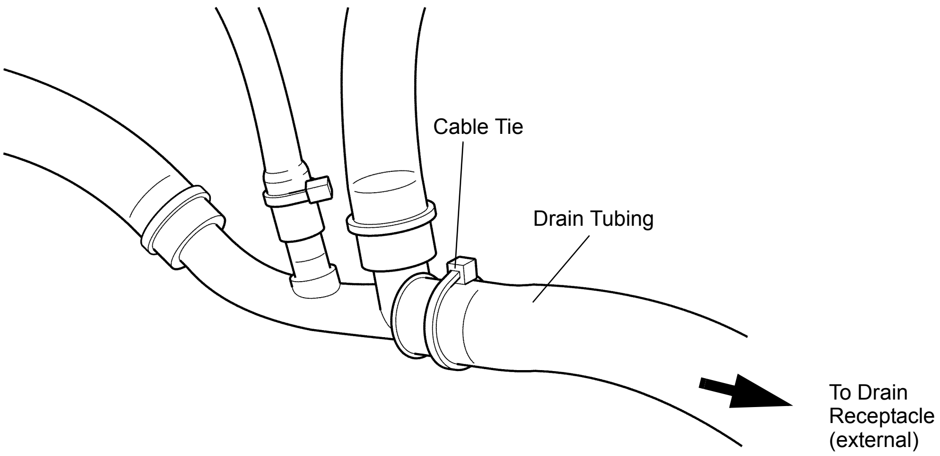


Figure 6.7 Connect the Drain Tubing

The drain tube from the dehumidifier drain port is located inside the right panel. Connect it to the external drain tubing using two of the provided cable ties to connect the drain tubing so that there are no leaks.

⚠ CAUTION

Insufficient drainage could cause an overflow of drainage liquid into the instrument.

Keep resistance to drainage flow to a minimum as follows.

- Ensure that the height of the drain tubing is never higher than the drain port on the instrument.
- Keep the tip of the drain tubing above the surface of the liquid in the drain container.
- Corrosive substances, such as acids, may be contained in the drainage. Be careful to avoid touching or spilling these liquids.

6.8.3.5 PC Cable Connections

This section describes the procedure for connecting the TOC-V to the PC using the supplied RS-232C cable.

Procedure

1. Insert the supplied RS-232C cable into the RS-232C connector (1) on the back of the TOC-V, and tighten the retaining screws on both sides. See Figure 2.4 "Rear View" for more details.

2. Insert the plug at the other end of the RS-232C cable into the RS-232C connector of the PC, and tighten the retaining screws on both sides.

6.8.4 Installing the Autosampler

6.8.4.1 Installing the Autosampler

This section describes the installation of the ASI-V autosampler.

Positioning the Autosampler

As shown in Figure 6.8 "Installing the Autosampler", the autosampler is placed to the left of the TOC-V.

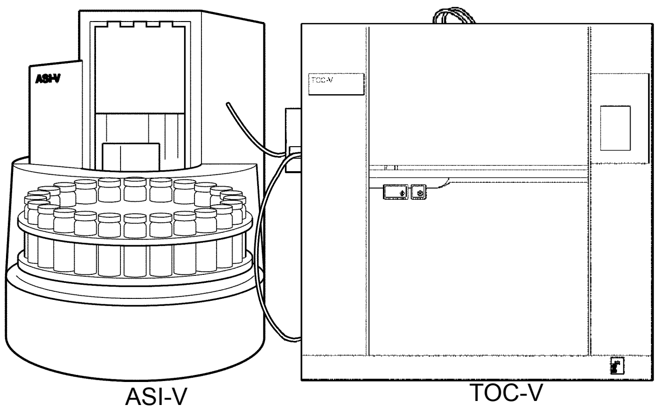


Figure 6.8 Installing the Autosampler

Note: Keep the distance between the autosampler and main unit to a minimum to allow a free range of motion of the autosampler arm. If the autosampler tubing is stretched, it may impede the movement of the autosampler arm, resulting in operation errors and damage.

Removing the Shipping Screw

The arm of the ASI is secured with a shipping screw to avoid damage during shipment. Before connecting power to the instrument, remove the ASI cover to access the shipping screw located on the retaining bracket. Remove the shipping screw and replace the cover. Label and retain the shipping screw.

CAUTION

To avoid instrument damage, the customer should verify that the ASI arm is firmly attached to the retaining bracket with the shipping screw before shipping the instrument or moving it to another location.

Connecting Cables

The TOC-V and ASI-V are connected using cables, as described below.

CAUTION

Verify that the TOC-V is switched OFF before performing the following cabling procedure.

Procedure

1. Connect the ASI-V power cord to the rear of the TOC-V.

2. Connect the provided signal cable to the ASI-V signal connectors at the rear of the TOC-V and the ASI-V.

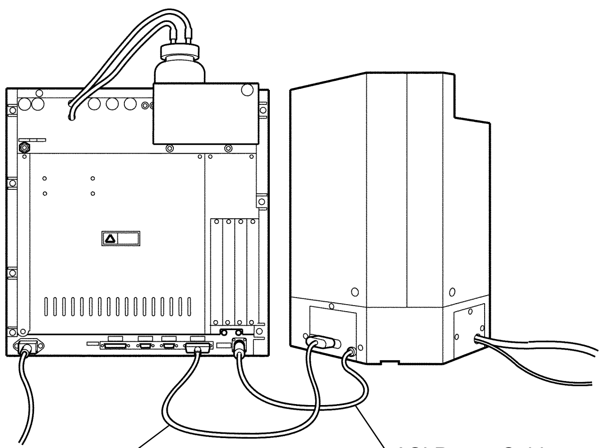


Figure 6.9 Connecting the Cables (TOC-V to ASI-V)

Connecting the Sample Tubing and Sparge Tubing (External Sparge Kit)

The procedure for connecting the ASI-V sample tubing and sparge tubing for the External Sparge Kit is as follows.

Procedure

1. Open the TOC-Control V Sample Table Editor and connect the instrument.

2. From the Instrument menu, select Maintenance>ASI Needle Change.

3. Click the Preparation Start button.
   The autosampler arm moves to the front of the needle replacement window, allowing easy access.

4. Remove the screw on the lower left side of the needle replacement window, and detach the window cover by pulling downward.

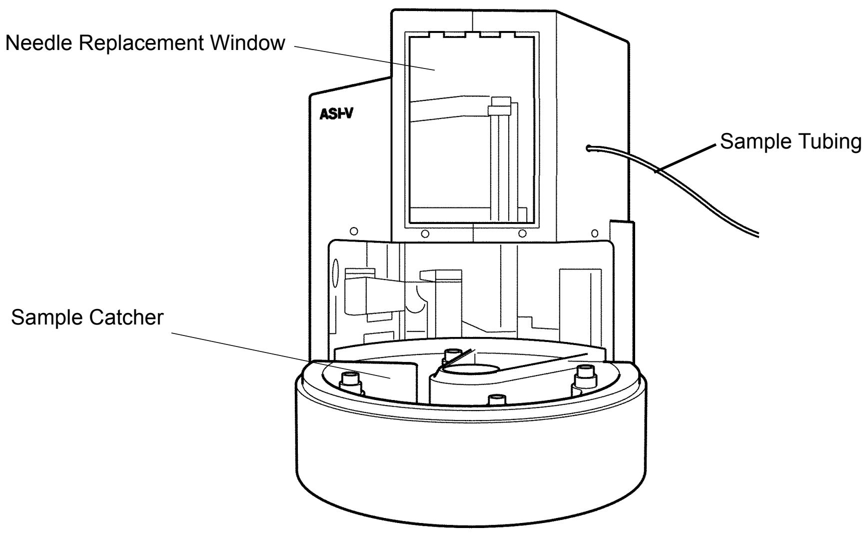


Figure 6.10 Tubing Installation

5. Install the bushing at the end of the sample tubing to the Number 2 port of the TOC-V sample injector 8-port valve.

6. Draw the sparge tubing out through the joint and connect the sparge tubing.

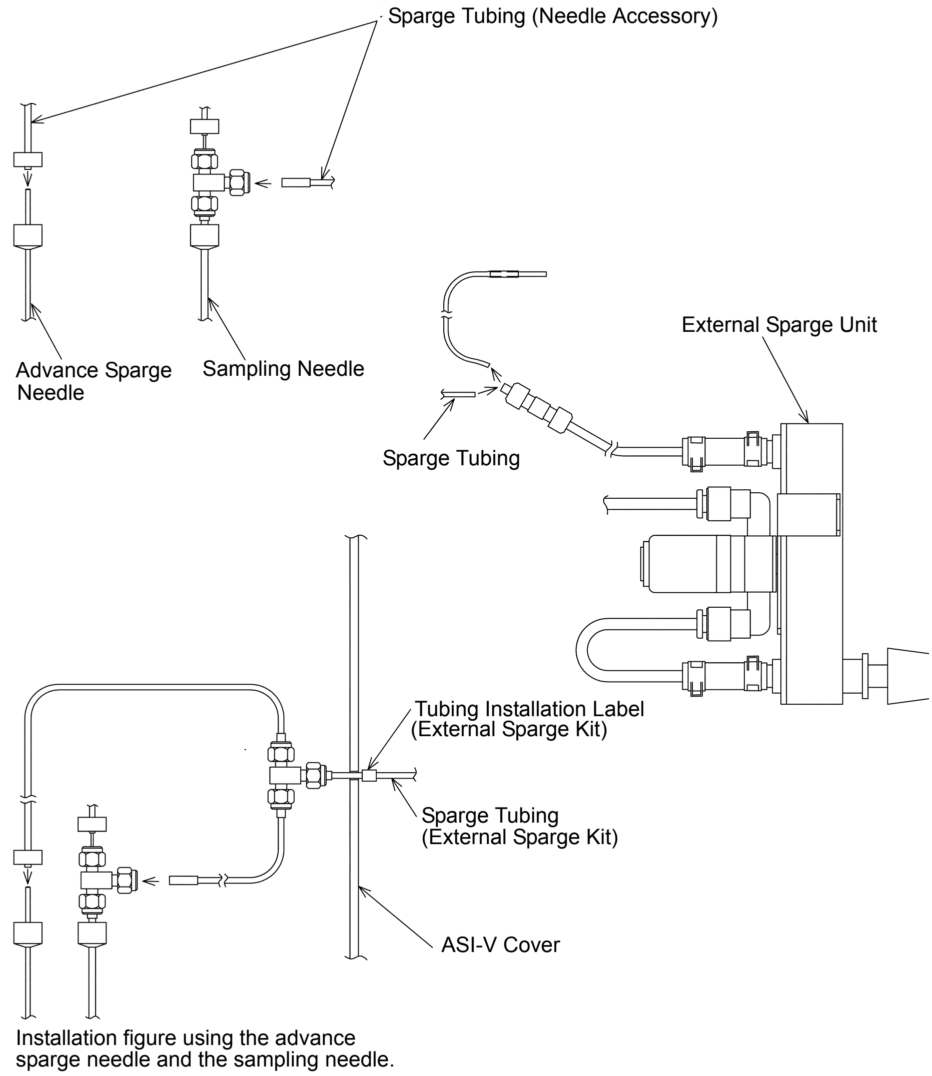


Figure 6.11 Installing the External Sparge Kit

Note: Do not use the flow controller knob to turn off the sparge gas flow.

Installing the Needle

This section describes installation of the ASI-V needle. There are two types of needles, the sample needle (same as the sparge needle) and the sample + sparge needle.

When installing the needle, remove the needle replacement window cover.

Installation Procedure for the Sample Needle and Sparge Needle

1. Open the TOC-Control V Sample Table Editor and connect the instrument.

2. From the Instrument menu, select Maintenance>ASI Needle Change.

3. Click the Preparation Start button.
   The autosampler arm moves to the front of the needle replacement window, allowing easy access.

4. Remove the screw, and then detach the needle holder from the arm.

5. Insert the needle into the opening in the needle holder.

6. Secure the needle by turning the retaining bracket and tightening the screw at the top of the needle holder.

   Note: There are two openings; the left opening is for the sample tubing and the right is for the sparge tubing.

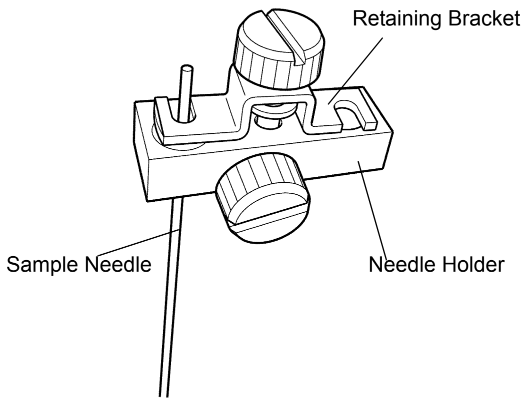


Figure 6.12 Installing the Needle - 1

7. Securely fit the sample tubing or sparge tubing onto the upper end of the needle.

   Note: Verify that the bushing is in position to secure the sample tubing onto the sample needle.

8. Insert the lower tip of the needle into the needle guide opening, thrusting the needle holder into the pin of the arm, then tighten the screw.

9. Re-install the needle replacement, window cover, and tighten the screw.

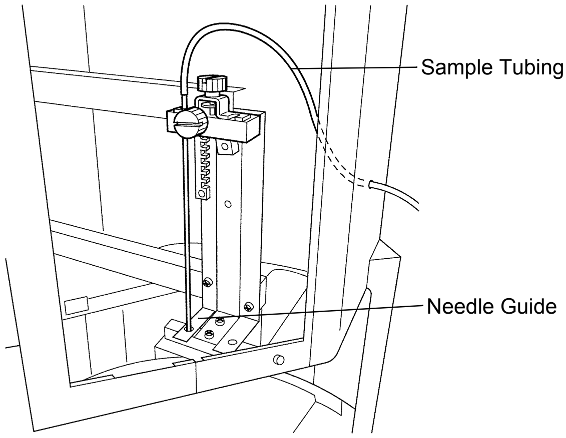


Figure 6.13 Installing the Needle - 2

Installing the Turntable, Vial Rack and Cover

Installing the Turntable

1. Place the turntable on the ASI-V.

Note: There is no distinction between front and rear of the turntable, so orientation is irrelevant.

2. Place the turntable so that the 3 guides on the ASI-V pass through the 3 holes at the center of the turntable.

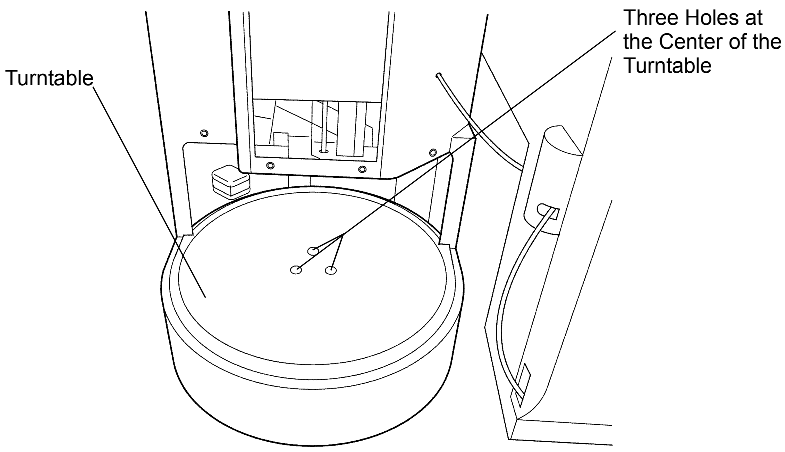


Figure 6.14 Installing the Turntable

Installing the Vial Rack

1. Place the vial rack on top of the turntable.

2. Rotate the vial rack until the guides of the turntable fit into the vial rack. This secures the vial rack in place.

Installing the Cover

1. Place the cover on the ASI-V.

   Note: There is a magnet at the left rear of the cover. Verify that the cover and ASI-V are in contact at the magnet.

Install the Rinse Bottle and Drain Tank

Installation of the rinse bottle and drain tank is explained below.

Rinse and drain tubing are attached to the autosampler, and are respectively labeled “RINSE” and “DRAIN.” Use the rinse water tubing as it is, and cut the drain tubing to an appropriate length.

⚠ CAUTION

Verify that the drain tubing remains lower than the drain port of the ASI-V, and that the end of the drain tubing remains above the surface of the liquid in the drain tank.

Corrosive substances, such as acids or etc., may be contained in the drainage. Avoid touching or spilling these liquids.

Preparation

- The rinse bottle is provided as a standard accessory.
- The drain bottle (P/N 638-52344) is provided as a special accessory. A suitable substitute may be used as the drain container.

Procedure

1. Place the rinse bottle on the bench with the TOC-V and the autosampler.
2. Place the drain container on the floor.
3. To minimize resistance, keep the tip of the drain tubing above the surface of the drain liquid and as close to the instrument as possible.

⚠ CAUTION

Do not allow any slack or bends in the tubing that could cause a buildup of water pressure.

Before Starting Analysis

Check the following before starting analysis.

- The rinse water bottle is filled to above the 2L mark on the bottle.
- The tip of the rinse tube reaches nearly to the bottom of the rinse water bottle.

⚠ CAUTION

Rinse water is not delivered and air may be taken into the TOC-V injection pump if the above checks are not made.

6.8.5 OCT-1 8-Port Sampler Installation

The OCT-1 8-port sampler is installed as described below.

6.8.5.1 Installation Procedure

This section describes the procedure for installing the OCT-1 8-Port Sampler.

The unit is set up to the left of the TOC-V main body as shown in Figure 6.15 "Installation of the 8-Port Sampler".

TIP: *When 2 OCT-1 8-Port Samplers are to be used, place both units to the left of the TOC-V main unit as shown in the overhead view of Figure 6.4 "External Dimensions with OCT-1 8-Port Sampler".*

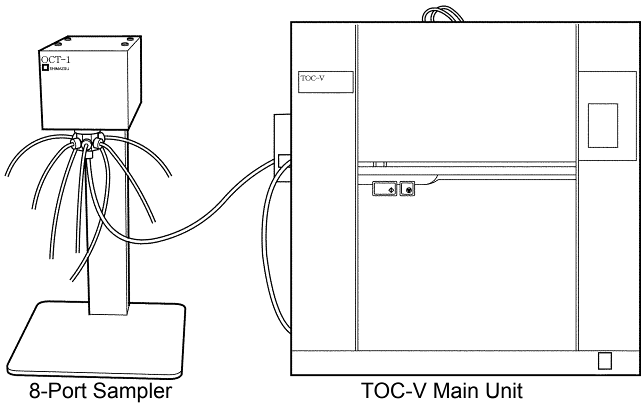


Figure 6.15 Installation of the 8-Port Sampler

Cable Connections

The cable is connected between the main unit of the TOC-V and the 8-Port Sampler.

CAUTION

Confirm that the power to the TOC-V main unit is switched OFF before performing this procedure.

Connection Procedure

1. Remove the 4 screws at the top of the 8-Port Sampler, and take off the cover.

2. Connect the power and signal cable to the circuit board of the 8-Port Sampler.

3. Connect the ground wire of the power cable to the grounding terminal on the circuit board of the OCT-1.

4. Pass the cable through the rear panel as shown in figure 1.8, and replace the cover of the 8-Port Sampler.

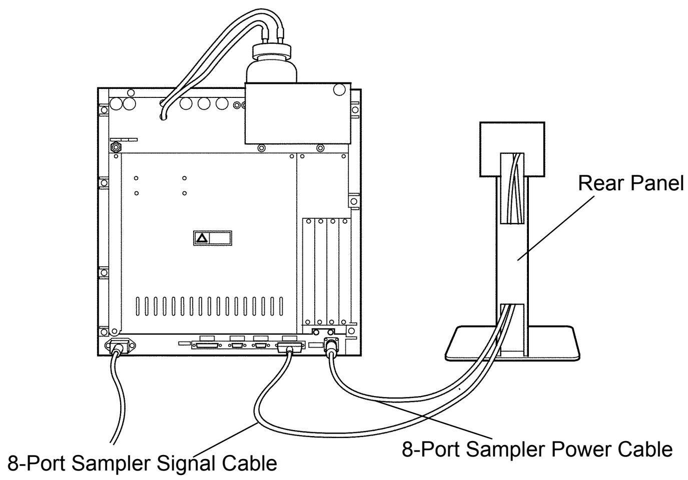


Figure 6.16 Cable Connections (TOC-V and 8-Port Sampler)

5. Connect the power cable to the power receptacle on the back of the TOC-V.

6. Connect the signal cable to the 8-Port Sampler connection on the back of the TOC-V.

Installing Two 8-Port Sampler Units

When installing two 8-Port Sampler units, a signal cable (standard accessory) is connected between 8-Port Sampler 1 and 8-Port Sampler 2.

Installation Procedure

1. Remove the covers from both 8-Port Sampler units.

2. Connect the signal cable exiting 8-Port Sampler 2 to the connector of 8-Port Sampler 1.

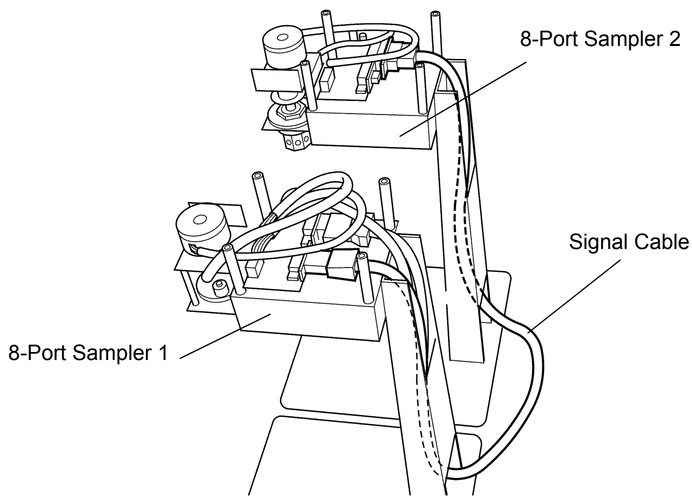


Figure 6.17 Connecting Two 8-Port Samplers

3. Remove the port number label from the 8-Port Sampler 2 unit and replace it with the port number label provided with the unit.

   Note: The port that was originally labeled as port No. 1 should be labeled as port No. 9.

4. Replace the covers on the 8-Port Samplers.

Connecting the Sample Tubing

Use the following procedure to connect the sample tubing of the 8-Port Sampler.

Tubing Connection Procedure

1. The sample tubing differs according to the TOC-V model that is used with the 8-Port Sampler

   Note: Refer to Table 6.15 "Maintenance Parts List 3 (OCT-1 8-Port Sampler)" for the appropriate part numbers.

2. Connect the fitting at one end of the tubing to the common (COM) port of the 8-Port Sampler.

3. Pass the sample tubing through the hole in the left side panel of the TOC-V main unit. Connect the fitting at the other end of the sample tubing to port No. 2 of the 8-port valve of the TOC-V sample injector. Connect to port No. 1 when installing 8-Port Sampler 2.

4. Connect the sample tubing to ports 1 - 8 (ports 9 - 16 for 8-Port Sampler 2) of the 8-Port Sampler.

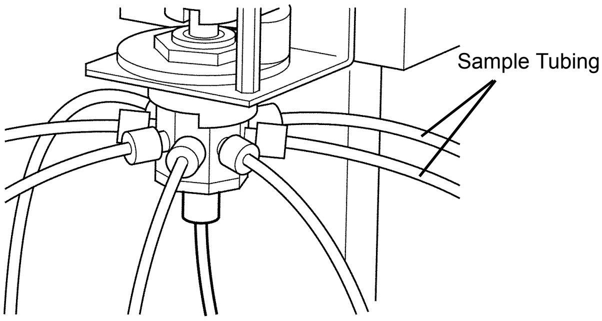


Figure 6.18 Tubing Connections

Note: Connection to the TOC-V Main Unit

- 8-Port Sampler 1

  Connect the COM tubing to the common (COM) port of 8-Port Sampler 1 and port No. 2 on the TOC-V unit.

- 8-Port Sampler 2

  Connect the COM tubing to the common (COM) port of 8-Port Sampler 2 and port No. 1 on the TOC-V unit

6.9 Material Safety Data Sheets

6.9.1 Sodium Persulfate

Shimadzu Scientific Instruments, Inc.
7102 Riverwood Drive Columbia, MD 21046
Tel: 1(800) 477-1227

Control No.
Date

MATERIAL SAFETY DATA SHEET

SECTION I PRODUCT IDENTIFICATION

CAT. NO.: CAS NO.: 7775-27-1
NAME: SODIUM PERSULFATE OTHER NAME: Sodium Peroxydisulfate, Peroxydisulfuric Acid Disodium Salt
PRODUCT USE: Specifically for use in Shimadzu TOC instruments. Consult the appropriate instrument manual for proper use.

SECTION II PHYSICAL DATA

APPEARANCE AND ODOR: White crystalline powder
PHYSICAL PROPERTIES: Specific gravity: 2.400

SECTION III HAZARDS IDENTIFICATION

Oxidizing, irritant, contact with combustible material may cause fire. Irritating to eyes, respiratory system and skin. Possible sensitizer. Keep away from combustible material. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Wear suitable protective clothing.

SECTION IV STABILITY AND REACTIVITY

INCOMPATIBILITIES: Strong reducing agents, finely powdered metals, strong bases. Protect from moisture, alcohols.
HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS: Toxic fumes of sulfur oxides.

SECTION V FIRE FIGHTING MEASURES

EXTINGUISHING MEDIA: Water spray.
SPECIAL FIREFIGHTING PROCEDURES: Wear self-contained breathing apparatus and protective clothing to prevent contact with skin and eyes. Strong oxidizer.
UNUSUAL FIRE AND EXPLOSIONS HAZARDS: Contact with other material may cause fire. Emits toxic fumes under fire conditions. Container explosion may occur under fire conditions.

SECTION VI TOXICITY DATA

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.
RTECS #: SE0525000 Peroxydisulfuric Acid, Disodium Salt
TOXICITY DATA: IPR-MUS LD50:226 mg/kg

SECTION VII HEALTH HAZARD DATA

Acute effects: May be harmful by inhalation, ingestion, or skin absorption. Causes eye and skin irritation. Material is irritating to mucous membranes and upper respiratory tract. Prolonged or repeated exposure may cause allergic reactions in certain sensitive individuals.

SECTION VIII FIRST-AID MEASURES

In case of contact, immediately flush eyes or skin with copious amounts of water for at least 15 minutes while removing contaminated clothing and shoes. Assure adequate flushing of the eyes by separating the eyelids with fingers. If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen. If swallowed, wash out mouth with water provided person is conscious. Call a physician. Wash contaminated clothing before reuse. Discard contaminated shoes.

SECTION IX ACCIDENTAL RELEASE MEASURES

Evacuate area. Shut off all sources of ignition. Wear self-contained breathing apparatus, rubber boots and heavy rubber gloves. Cover with dry-lime, sand, or soda ash. place in covered containers using non-sparking tools and transport outdoors. Ventilate area and wash spill site after material pickup is complete.

SECTION X SPECIAL PRECAUTIONS AND COMMENTS

The above information is believed to be correct but does not purport to be all inclusive. It shall serve only as a guide. Shimadzu shall not be held liable for any damage resulting from handling or contact with the above product. Users should make their own determinations regarding the suitability of this information for their particular purposes.

6.9.2 Phosphoric Acid

Shimadzu Scientific Instruments, Inc.
7102 Riverwood Drive Columbia, MD 21046
Tel: 1(800) 477-1227

Control No.
Date

MATERIAL SAFETY DATA SHEET

SECTION I **PRODUCT IDENTIFICATION**

CAT NO.: 630-00710-00 CAS NO.: 07664-38-2
NAME: Phosphoric Acid 25% Ultrapure OTHER NAME: Phosphoric Acid, IC Reaction Liquid

SECTION II **PRDUCT USE**

This product is specifically for use in the IC channel of the TOC 500, TOC 5000, and TOC 4000 series of TOC analyzers. Refer to the appropriate instruction manual for directions on proper use. Keep tightly sealed in a cool, dry, well ventilated place. Store separate form flammable and combustible materials. Wear appropriate protective clothing and eye protection.

SECTION III **PHYSICAL DATA**

Melt Point	Boil Point	Density	Vapor Pressure	Color	Phase	Water Solubility
N/A	N/A	N/A	N/A	Colorless / Clear	Liquid	Completely Soluble

SECTION IV **HAZARDS IDENTIFICATION**

Corrosive, causes burns. Harmful if swallowed. Target organ(s): liver, blood. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Take off immediately all contaminated clothing. Wear suitable protective clothing, gloves and eye/face protection.

SECTION V **TOXICITY DATA**

UNR-MAN LDLO:	220 mg/kg	ORL-RAT LD50:	1530 mg/kg
IHL-RAT LC50:	>850 mg/m3/1h	SKN-RBT LD50:	2740 mg/kg

SECTION VI **HEALTH HAZARD DATA**

Acute Effects: Harmful if swallowed. May be harmful if inhaled. May be harmful if absorbed through the skin. Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes and skin. Inhalation may result in spasm, inflammation and edema of the larynx and bronchi, chemical pneumonitis and pulmonary edema. Symptoms of exposure may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. May cause cyanosis.
Chronic Effects: Target organ(s): liver, blood, bone marrow. To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

SECTION VII **FIRST-AID MEASURES**

In case of contact, immediately flush eyes or skin with copious amounts of water for at least 15 minutes while removing contaminated clothing and shoes. Assure adequate flushing of the eyes by separating the eyelids with fingers. If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen. If swallowed, wash out mouth with water provided person is conscious. Call a physician. Wash contaminated clothing before reuse. Discard contaminated shoes.

SECTION VIII **FIRE FIGHTING MEASURES**

EXTINGUISHING MEDIA: Carbon dioxide, dry chemical powder or appropriate foam.
SPECIAL FIREFIGHTING PROCEDURES: Wear self-contained breathing apparatus and protective clothing to prevent contact with skin and eyes.
UNUSUAL FIRE AND EXPLOSIONS HAZARDS: Emits toxic fumes under fire conditions.

SECTION IX **ACCIDENTAL RELEASE MEASURES**

Evacuate area. Wear self-contained breathing apparatus, rubber boots and heavy rubber gloves. Cover with dry lime or soda ash, pick up, keep in a closed container and hold for waste disposal. Ventilate area and wash spill site after material pickup is complete.

SECTION X **STABILITY AND REACTIVITY**

INCOMPATIBILITIES: Strong bases, finely powdered metals
HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS: Thermal decomposition may produce toxic fumes of phosphorus oxides and/or phosphine.

SECTION XI **SPECIAL PRECAUTIONS AND COMMENTS**

The above information is believed to be correct but does not purport to be all inclusive. It shall serve only as a guide. Shimadzu shall not be held liable for any damage resulting from handling or contact with the above product. Users should make their own determinations regarding the suitability of this information for their particular purposes.

6.9.3 CO_2 Absorber, $Ca(OH)_2$, KOH, NaOH: Soda Lime

Shimadzu Scientific Instruments, Inc.
7102 Riverwood Drive Columbia, MD 21046
Tel: 1(800) 477-1227

Control No.
Date

MATERIAL SAFETY DATA SHEET

SECTION I **PRODUCT IDENTIFICATION**

CAT. NO. 630-00556-00 CAS NO. 80006-28-8
NAME: Soda Lime OTHER NAME: Soda Lime; Soda and Lime
PRODUCT USE: CO_2 Scrubber in Shimadzu TOC Analyzers

SECTION II **PRECAUTIONS TO BE TAKEN IN HANDLING**

Protect skin, eyes and clothing. Avoid prolonged or repeated exposure. Wash thoroughly after handling. Safety shower and eye bath. Use 8" minimum face shield. Store as a corrosive. Keep tightly closed and store in a cool, dry, place. Air and moisture sensitive. Use as directed in the TOC-4000 Instruction Manual.

SECTION III **PHYSICAL DATA**

Melt Point	Boil Point	Density	Vapor Pressure	Vapor Density	Evaporation Rate	Odor	Color	Phase	Water Solubility
N/A	N/A	N/A	N/A	N/A	N/A	N/A	White	Granules, various sizes	N/A

SECTION IV **REACTIVITY DATA**

Incompatible with strong acids. Absorbs CO_2, moisture inactivates.

SECTION V **FIRE AND EXPLOSION HAZARD DATA**

EXTINGUISHING MEDIA: Dry chemical fire extinguisher. Wear self-contained breathing apparatus and protective clothing to prevent skin and eye exposure.
UNUSUAL FIRE HAZARD: Emits toxic fumes in a fire.

SECTION VI **TOXICITY DATA**

RAT/MOUSE LD50	RTECS#	OSHA PEL	ACGIH TLV-TWA
N/A	VX965000	N/A	N/A

SECTION VII **HEALTH HAZARD DATA**

Acute effects: Harmful if swallowed, inhaled, or absorbed through skin. Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes and skin. Inhalation may be fatal as a result of spasm, inflammation and edema of the larynx and bronchi, chemical pneumonitis and pulmonary edema. Symptoms of exposure may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

SECTION VIII **FIRST AID**

An antidote is a substance intended to counteract the effect of a poison. It should be administered only by a physician or trained emergency personnel. Medical advice can be obtained from a POISON CONTROL CENTER.
In case of contact: Immediately flush eyes or skin with copious amounts of water for at least 15 minutes while removing contaminated clothing and shoes. Assure adequate flushing of the eyes by separating eyelids with fingers. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. If swallowed, wash out mouth with water provided person is conscious. CALL A PHYSICIAN. Wash contaminated clothing before reuse. Discard contaminated shoes. For complete information, see actual entry in RTECS #VX965000.

SECTION IX **SPILL OR LEAK PROCEDURES**

Occupational spill: Evacuate area. Wear self-contained breathing apparatus, rubber boots and heavy rubber gloves. Absorb on sand or vermiculite and place in closed containers for disposal. Ventilate area and wash spill site after material pickup is complete.

SECTION X **SPECIAL PRECAUTIONS AND COMMENTS**

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. SHIMADZU shall not be held liable for any damage resulting from handling or from contact with the above product. Users should make their own investigations to determine the suitability of the information for their particular purposes.

6.9.4 Halogen Scrubber

Shimadzu Scientific Instruments, Inc.
7102 Riverwood Drive Columbia, MD 21046
Tel: 1(800) 477-1227

Control No.
Date

MATERIAL SAFETY DATA SHEET

SECTION I PRODUCT IDENTIFICATION

CAT. NO. 630-0092-00 CAS NO.07740-50-8
NAME: Halogen Scrubber OTHER NAME:Copper Wool
PRODUCT USE: This product is for specific use with TOC instruments.

SECTION II PHYSICAL DATA

Melt Point	Boil Point	Density	Vapor Pressure	Vapor Density	Evaporation Rate	Odor	Color	Phase	Water Solubility
1083°C	2595°C	N/A	N/A	N/A	N/A	N/A	Reddish	Solid Malleable Metal	N/A

SECTION III HAZARDS IDENTIFICATION

Dust, mist or fumes may cause eye and skin irritation. Avoid contact.

SECTION IV TOXICITY DATA

RAT/MOUSE LD50	RTECS#	OSHA PEL	ACGIH TLV-TWA
N/A	GL5325000	N/A	N/A

SECTION V HEALTH HAZARD DATA

Exposure to large amounts can cause gastrointestinal disturbances, nausea, vomiting, headaches, or dizziness.

SECTION VI FIRST-AID MEASURES

In case of contact, immediately flush eyes with copious amounts of water for at least 15 minutes. in case of skin contact, immediately wash skin with soap and copious amounts of water. if ingested and subject is conscious, immediately give large amounts of water. get medical attention.

SECTION VII FIRE FIGHTING MEASURES

EXTINGUISHING MEDIA: Appropriate to surrounding fire. Possible formation of toxic copper fumes. Use appropriate respiratory support.

SECTION VII ACCIDENTAL RELEASE MEASURES

N/A

SECTION IX STABILITY AND REACTIVITY

INCOMPATIBILITIES: Strong acids, active halogens such as chlorine, fluorine, iodine, and bromine. ammonia.

SECTION X SPECIAL PRECAUTIONS AND COMMENTS

The above information is believed to be correct but does not purport to be all inclusive. It shall serve only as a guide. Shimadzu shall not be held liable for any damage resulting from handling or contact with the above product. Users should make their own determinations regarding the suitability of this information for their particular purposes.

6.9.5 Sodium Bicarbonate

Shimadzu Scientific Instruments, Inc.
7102 Riverwood Drive Columbia, MD 21046
Tel: 1(800) 477-1227

Control No.
Date

MATERIAL SAFETY DATA SHEET

SECTION I **PRODUCT IDENTIFICATION**

CAT. NO. 630-00963-01 CAS NO. 144-55-8
NAME Sodium Bicarbonate 100% OTHER NAME: Sodium hydrogen carbonate, Baking soda

SECTION II **PRDUCT USE**

This product is specifically for use as a standard in TOC. Refer to the appropriate instruction manual for directions on proper use.

SECTION III **PHYSICAL DATA**

Melt Point	Boil Point	Density	Vapor Pressure	Vapor Density	Evaporation Rate	Odor	Color	Phase	Water Solubility
50°C	N/A	2.16	N/A	N/A	N/A	N/A	White	Solid Powders	10%

SECTION IV **HAZARDS IDENTIFICATION**

Possible eye and skin irritation. Avoid contact.

SECTION V **TOXICITY DATA**

N/A

SECTION VI **HEALTH HAZARD DATA**

Exposure to large amounts can cause gastrointestinal disturbances.

SECTION VII **FIRST-AID MEASURES**

In case of contact, immediately flush eyes with copious amounts of water for at least 15 minutes. In case of skin contact, immediately wash skin with soap and copious amounts of water.

SECTION VIII **FIRE FIGHTING MEASURES**

N/A

SECTION IX **ACCIDENTAL RELEASE MEASURES**

N/A

SECTION X **STABILITY AND REACTIVITY**

N/A

SECTION XI **SPECIAL PRECAUTIONS AND COMMENTS**

The above information is believed to be correct but does not purport to be all inclusive. It shall serve only as a guide. Shimadzu shall not be held liable for any damage resulting from handling or contact with the above product. Users should make their own determinations regarding the suitability of this information for their particular purposes.

6.9.6 Sodium Carbonate

Shimadzu Scientific Instruments, Inc.
7102 Riverwood Drive Columbia, MD 21046
Tel: 1(800) 477-1227

Control No.
Date

MATERIAL SAFETY DATA SHEET

SECTION I **PRODUCT IDENTIFICATION**

CAT. NO. 630-0092-01 CAS NO. 497-19-8
NAME: Sodium Carbonate, 100% OTHER NAME: N/A

SECTION II **PRDUCT USE**

This product is specifically for use in the IC channel of the TOC 500, TOC 5000, and TOC 4000 series of TOC analyzers. Refer to the appropriate instruction manual for directions on proper use. Keep tightly sealed in a cool, dry, well ventilated place. Store separate form flammable and combustible materials. Wear appropriate protective clothing and eye protection.

SECTION III **PHYSICAL DATA**

Melt Point	Boil Point	Density	Vapor Pressure	Vapor Density	Evaporation Rate	Odor	Color	Phase	Water Solubility
N/A	N/A	2.16	N/A	N/A	N/A	N/A	White	Solid Powder or Crystals	Completely Soluble

SECTION IV **HAZARDS IDENTIFICATION**

Corrosive, causes burns, harmful if swallowed. Target organ(s): liver, blood. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Take off all contaminated clothing. Wear suitable protective clothing, gloves and eye/face protection.

SECTION V **TOXICITY DATA**

ORL-RAT LD50:4090 MG/KG,IHL-RAT LC50:2300 MG/M3/2H
ORL-MUS LD50:6600 MG/KG,IHL-MUS LC50:1200 MG/M3/2H
SCU-MUS LD50:2210 MG/KG,IHL-GPG LC50:800 MG/M3/2H

SECTION VI **HEALTH HAZARD DATA**

Harmful if swallowed, inhaled, or absorbed through skin.causes severe irritation. High concentrations are extremely destructive to tissues of the mucous membranes and upper respiratory tract, eyes and skin. Symptoms of exposure may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

SECTION VII **FIRST-AID MEASURES**

In case of contact, immediately flush eyes or skin with copious amounts of water for at least 15 minutes while removing contaminated clothing and shoes. Assure adequate flushing of the eyes by separating the eyelids with fingers. If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen. If swallowed, wash out mouth with water provided person is conscious. Call a physician. wash contaminated clothing before reuse. Discard contaminated shoes.

SECTION VIII **FIRE FIGHTING MEASURES**

NONCOMBUSTIBLE. Use extinguishing media appropriate to surrounding fire conditions.
SPECIAL. Wear self-contained breathing apparatus and protective clothing to prevent contact with skin and eyes.
UNUSUAL FIRE AND EXPLOSIONS HAZARDS.Emits toxic fumes under fire conditions.

SECTION IX **ACCIDENTAL RELEASE MEASURES**

Evacuate area. Wear self-contained breathing apparatus, rubber boots and heavy rubber gloves. Wear disposable coveralls and discard them after use. Sweep up, place in a bag and hold for waste disposal. Avoid raising dust.Ventilate area and wash spill site after material pickup is complete.

SECTION X **STABILITY AND REACTIVITY**

INCOMPATIBILITIES: Strong acids, aluminum, protect from moisture.
HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS: Carbon monoxide, carbon dioxide.

SECTION XI **SPECIAL PRECAUTIONS AND COMMENTS**

The above information is believed to be correct but does not purport to be all inclusive. It shall serve only as a guide. Shimadzu shall not be held liable for any damage resulting from handling or contact with the above product. Users should make their own determinations regarding the suitability of this information for their particular purposes.

6.9.7 Potassium Hydrogen Phthalate

Shimadzu Scientific Instruments, Inc.
7102 Riverwood Drive Columbia, MD 21046
Tel: 1(800) 477-1227

Control No.
Date

MATERIAL SAFETY DATA SHEET

SECTION I **PRODUCT IDENTIFICATION**

CAT. NO. 630-00635-01 CAS NO. 877-24-7
NAME: Potassium Hydrogen Phthalate OTHER NAME: Phthalic Acid, Monopotassium Salt, Khp Primary Standard

SECTION II **PRODUCT USE**

Specifically for use as an organic carbon standard in the Shimadzu TOC 500, TOC 5,000, and TOC 4,000 instruments. Consult the appropriate instrument manual for proper use.

SECTION III **PHYSICAL DATA**

Melt Point	Boil Point	Density	Vapor Pressure	Vapor Density	Evaporation Rate	Odor	Color	Phase	Water Solubility
295-300°C	N/A	N/A	N/A	N/A	N/A	N/A	White	Crystals	N/A

SECTION IV **HAZARDS IDENTIFICATION**

Irritant, irritating to eyes, respiratory system and skin. Possible sensitizer. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Wear suitable protective clothing.

SECTION V **TOXICITY DATA**

RTECS #: CZ4326000
1,2-BENZENEDICARBOXYLIC ACID, MONOPOTASSIUM SALT
ORL-RAT LD50:>3200 MG/KG KODAK* 21MAY1971
SKN-GPG LD50:>1 GM/KG KODAK* 21MAY1971
Only selected registry of toxic effects of chemical substances (RTECS) data is presented here. See actual entry in RTECS for complete information.

SECTION VI **HEALTH HAZARD DATA**

ACUTE EFFECTS: May be harmful by inhalation, ingestion, or skin absorption; causes skin irritation; vapor or mist is irritating to the eyes, mucous membranes and upper respiratory tract.
EXPOSURE CAN CAUSE ASTHMATIC SYMPTOMS: Cough, hoarseness, rhinorrhea, wheezing, and conjunctivitis, nausea, or vomiting. Prolonged or repeated exposure may cause allergic reactions in certain sensitive individuals. To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

SECTION VII **FIRST-AID MEASURES**

In case of contact, immediately flush eyes or skin with copious amounts of water for at least 15 minutes while removing contaminated clothing and shoes. If inhaled, remove to fresh air. if not breathing give artificial respiration. If breathing is difficult, give oxygen. If swallowed, wash out mouth with water provided person is conscious. Call a physician. wash contaminated clothing before reuse.

SECTION VIII **FIRE FIGHTING MEASURES**

EXTINGUISHING MEDIA: Water spray, carbon dioxide, dry chemical powder or appropriate foam.
SPECIAL FIREFIGHTING PROCEDURES: Wear self-contained breathing apparatus and protective clothing to prevent contact with skin and eyes.
UNUSUAL FIRE AND EXPLOSIONS HAZARDS: Emits toxic fumes under fire conditions.

SECTION IX **ACCIDENTAL RELEASE MEASURES**

Wear respirator, chemical safety goggles, rubber boots and heavy rubber gloves. Sweep up, place in a bag and hold for waste disposal. Avoid raising dust. Ventilate area and wash spill site after material pickup is complete.

SECTION X **STABILITY AND REACTIVITY**

STABILITY: Stable.
INCOMPATIBILITIES: Strong oxidizing agent
HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS: Toxic fumes of carbon monoxide, carbon dioxide
HAZARDOUS POLYMERIZATION: Will NOT occur.

SECTION XI **SPECIAL PRECAUTIONS AND COMMENTS**

The above information is believed to be correct but does not purport to be all inclusive. It shall serve only as a guide. Shimadzu shall not be held liable for any damage resulting from handling or contact with the above product. Users should make their own determinations regarding the suitability of this information for their particular purposes.

6.9.8 Water

Shimadzu Scientific Instruments, Inc.
7102 Riverwood Drive Columbia, MD 21046
Tel: 1(800) 477-1227

Control No.
Date

MATERIAL SAFETY DATA SHEET

SECTION I PRODUCT IDENTIFICATION

CAT. NO. CAS NO. 7732-18-5
NAME: Water OTHER NAME: Dihydrogen Oxide
MOLECULAR WEIGHT: 18 CHEMICAL FORMULA: H_2O

SECTION II HAZARDS IDENTIFICATION

EMERGENCY OVERVIEW: Not applicable.
HEALTH RATING: 0 - None CONTACT RATING: 0 - None
FLAMMABILITY RATING:0 - None LAB PROTECTIVE EQUIP: Goggles; lab coat
REACTIVITY RATING: 1 - Slight STORAGE COLOR CODE: Orange (General Storage)

SECTION III HEALTH EFFECTS

POTENTIAL HEALTH EFFECTS:Water is non-hazardous.
INHALATION: Not applicable. EYE CONTACT: Not applicable.
INGESTION: Not applicable. CHRONIC EXPOSURE: Not applicable.
SKIN CONTACT Not applicable. AGGRAVATION OFPRE-EXISTING CONDITIONS: Not applicable.

SECTION IV FIRST AID MEASURES

NHALATION: Not applicable. SKIN CONTACT: Not applicable.
INGESTION: Not applicable. EYE CONTACT: Not applicable.

SECTION V FIRE FIGHTING MEASURES

FIRE: Not applicable. EXPLOSION: Not applicable.
FIRE EXTINGUISHING MEDIA:Use extinguishing media appropriate for surrounding fire.
SPECIAL INFORMATION: In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

SECTION VI ACCIDENTAL RELEASE MEASURES

Non-hazardous material. Clean up of spills requires no special equipment or procedures.

SECTION VII HANDLING AND STORAGE

Keep container tightly closed. Suitable for any general chemical storage area. Protect from freezing. Water is considered a non-regulated product, but may react vigorously with some materials. Avoid contact with all materials until investigation shows substance is compatible.

SECTION VII EXPOSURE CONTROLS/PERSONAL PROTECTION

AIRBORNE EXPOSURE LIMITS: Not applicable.
VENTILATION SYSTEM: Not applicable. PERSONAL RESPIRATORS (NIOSH APPROVED): Not applicable.
SKIN PROTECTION: None required. EYE PROTECTION: None required.

SECTION VIII PHYSICAL AND CHEMICAL PROPERTIES

Melt Point	Boil Point	Specific Gravity	Vapor Pressure	Vapor Density	Evaporation Rate	pH	Color	Phase	Water Solubility
0°C	100°C	1.00	17.5 @ 20°C	N/A	N/A	7.0	Clear, Colorless	Liquid	Complete

SECTION IX STABILITY AND REACTIVITY

STABILITY: Stable under ordinary conditions of use and storage.
HAZARDOUS DECOMPOSITION PRODUCTS: Not applicable.
HAZARDOUS POLYMERIZATION: Will not occur.
INCOMPATIBILITIES: Strong reducing agents, acid chlorides, phosphorus trichloride, phosphorus pentachloride, phosphorus oxychloride.

SECTION X ECOLOGICAL INFORMATION

ENVIRONMENTAL FATE: Not applicable.
ENVIRONMENTAL TOXICITY: Not applicable.
SECTION XIDISPOSAL CONSIDERATIONS: Whatever cannot be saved for recovery or recycling should be flushed to sewer. If material becomes contaminated during use, dispose of accordingly. Dispose of container and unused contents in accordance with federal, state and local requirements.

SECTION XII TRANSPORT INFORMATION

Not regulated.

SECTION XIII OTHER INFORMATION

NFPA RATINGS: Health: 0 Flammability: 0 Reactivity: 0 LABEL FIRST AID: Not applicable.
LABEL HAZARD WARNING: Not applicable. PRODUCT USE: Laboratory Reagent.
LABEL PRECAUTIONS: Keep in tightly closed container.

SECTION XIV SPECIAL PRECAUTIONS AND COMMENTS

The above information is believed to be correct but does not purport to be all inclusive. It shall serve only as a guide. Shimadzu shall not be held liable for any damage resulting from handling or contact with the above product. Users should make their own determinations regarding the suitability of this information for their particular purposes.

A

Appendix A

Method Validation

The Method Validation function tests the variance and the linearity of the data. The function also determines performance characteristics according to the requirements of DIN 38402-Part 51. The Method Validation function displays the data used for executing method validation, performs the required calculations and displays the results. Data can either be imported directly from the TOC-Control V Sample Table, or entered manually into the data table. Data can also be exported to ASCII files, stored and printed.

A.1 Main Window

The Main Window is separated into three sections. The upper left section of the window displays the calibration curve, the upper right section displays the calculated validation results and the lower portion displays the data table, which contains all relevant data for performing the validation. Refer to Section 4.4.2 "File Menu" and Section 4.4.3 "Edit Menu" for descriptions of the toolbar options, such as Open, Save, Cut, Copy, and Paste.

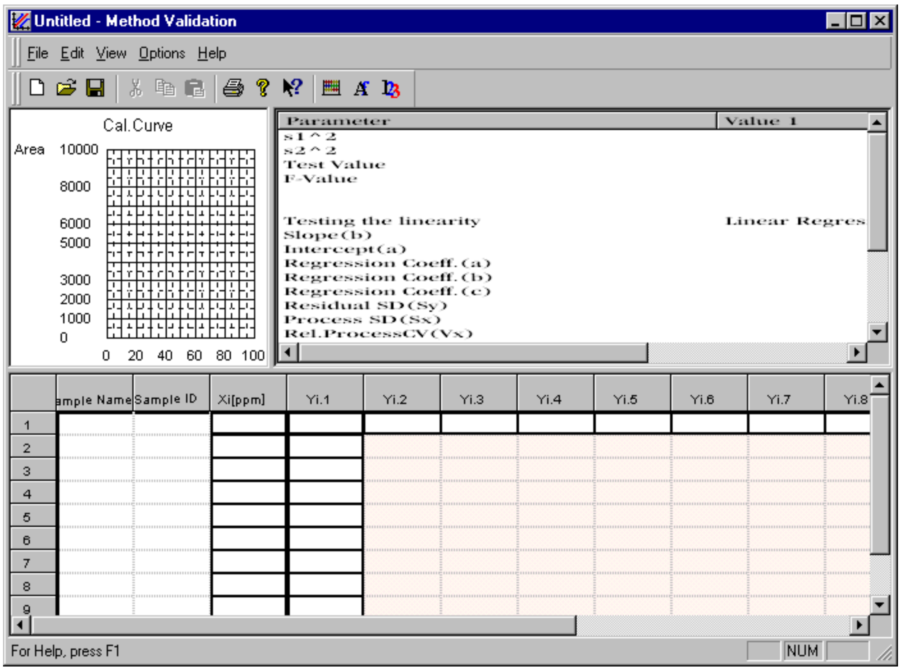


Figure A.1 Method Validation Main Window

Note: Windows can be resized by dragging the dividers between the sections.

A.2 File Menu

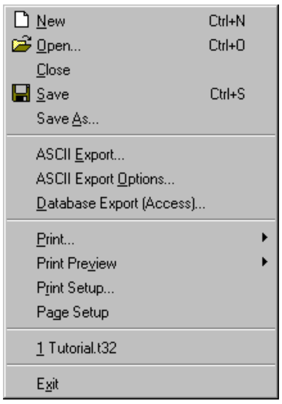


Figure A.2 File Menu

A.2.1 New

Use the New command to create a new, empty method validation file. A new file can also be created by clicking the New toolbar button. If a file that contains modified and unsaved data is open when the New command is selected, a prompt is displayed in a dialog box. Select one of the options in the dialog box to determine if the unsaved data should be saved before opening a new method validation file. Click the Yes button to save the document and create a new method validation file. Click the No button to create a new document without saving previous data.

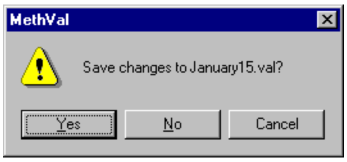


Figure A.3 Prompt for Saving Data

A.2.2 Open

Use the Open command to open a saved method validation file (*.val). A file can also be opened by clicking the Open toolbar button.

Start the method validation process by selecting a.cal file; application of DIN 38402 will begin. The process can also be started by selecting the calibration curve row in the Sample Table and opening the method validation function using the Tools>Method Validation command.

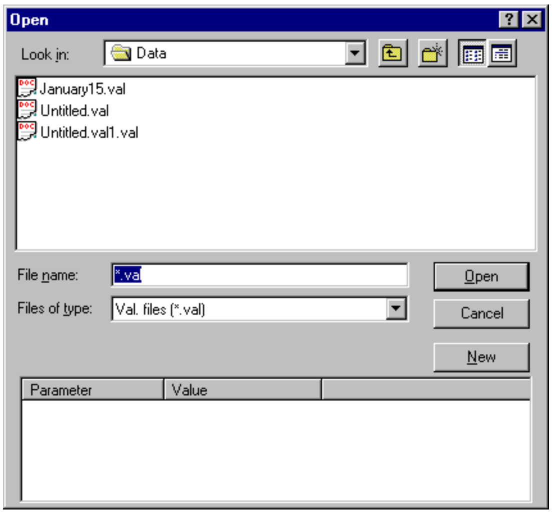


Figure A.4 Open Dialog Box

When the Open command is selected, the File>Open dialog box is displayed. To open an existing file, select the filename and choose the Open button. To create a new method validation file, type the filename into the File name text field, then select the New button. Refer to Section 4.4.2.4 “Open” for further information on the standard functions of the Open dialog box.

Note: An error message is displayed if a file that is not in a correct, TOC-Control V format is selected to be opened.

A.2.3 Save

Use the Save command to save data in the current active file. If the file has not yet been saved, selecting the Save command opens the Save As dialog box. The Save command can also be executed by clicking the Save toolbar button.

A.2.4 Save As

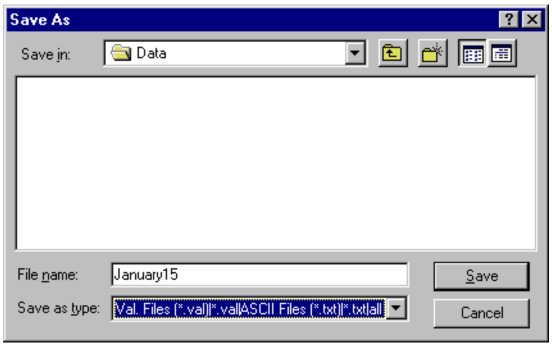


Figure A.5 Save As Dialog Box

The Save As command saves the active, current data to a new file name. Enter the desired name in the File Name field. Two different file types are available: validation files (*.val) used for storing the data in binary files and ASCII files (*.txt) used for exporting the data. Select the desired file type from the Save As Type drop-down list. Use the Save In window to select the location where the file should be saved. Refer to Section 4.4.2.7 “Save As” for details about the Save As command.

A.2.5 Print

Use the Print command to print data from a method validation file. Executing the Print command opens the Print dialog box; displayed options vary based on the installed printer driver. The Print command can also be executed by clicking the Print toolbar button.

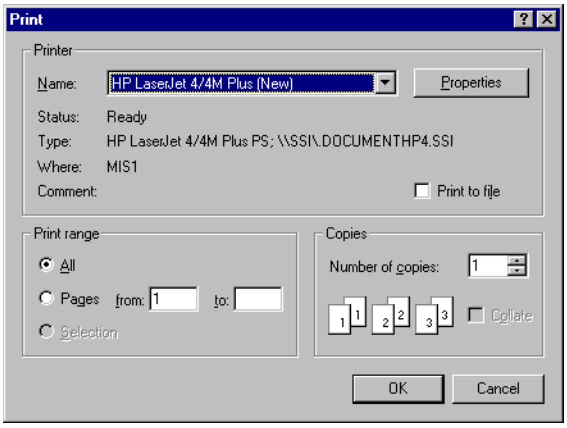


Figure A.6 Print Dialog Box

Use the Print Range option to specify the range to be printed. The All selection prints all pages in the file. Specific pages can be printed by selecting the Pages option and entering the desired page numbers. Use the Number of Copies field to select the number of copies to be printed.

A.2.6 Print Preview

Use the Print Preview command to view the method validation file before printing.

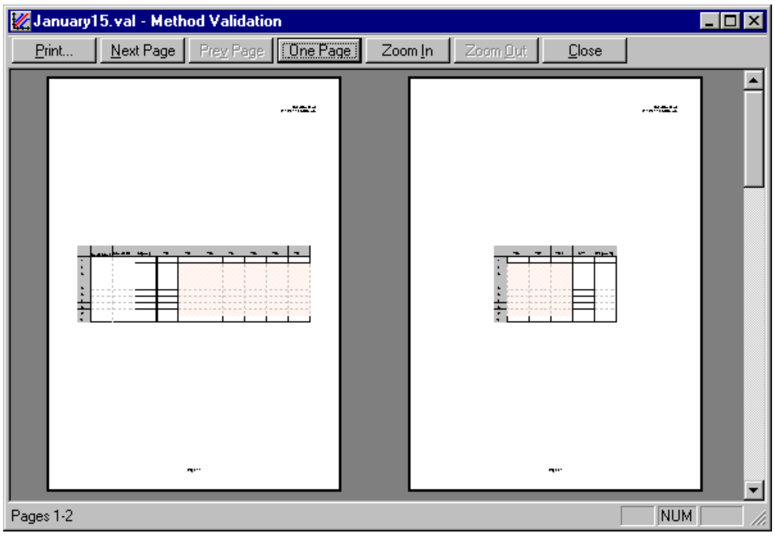


Figure A.7 Print Preview Window

Button	Description
Print	Click the Print button to print the report.
Next Page and Prev Page	Click the Next Page button to display the next page of the report. Click the Prev Page button to display the previous page of the report. The Next Page button is disabled when the last page of the report is displayed, and the Prev Page button is disabled when the first page of the report is displayed.
One Page/Two Page	Click the One Page button to display one page at a time in the Print Preview window. After selection, the button toggles to the Two Page button. Click the Two Page button to display two pages side by side.
Zoom In and Zoom Out	Click the Zoom In and Zoom Out buttons to change the magnification of the displayed pages. Three different zoom factors are available. Repeated selection of either option will continue to increase or decrease the magnification, as applicable.
Close	Click the Close button to close the Print Preview window and return to the previous window. The Print Preview window must be closed to edit the report.

A.2.7 Print Setup

Select Print Setup to open the Print Setup dialog box.

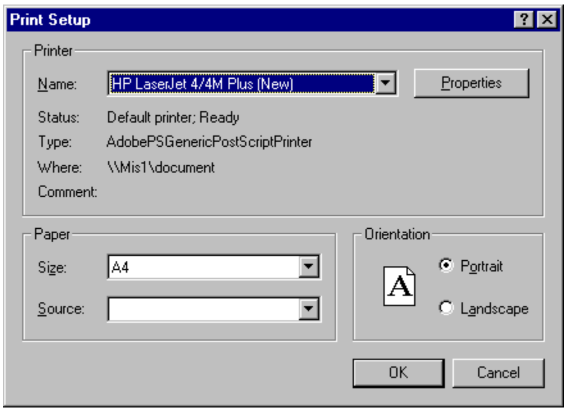


Figure A.8 Print Setup Dialog Box

The print options available vary based on the printer installed. Select the paper size and source from the drop-down lists, and select Portrait or Landscape to change the orientation of the printed pages. Click the OK button to save the selections, or click the Cancel button to close the dialog box without saving changes.

A.2.8 Page Setup

Use the Page Setup command to select items to be printed and to select options for printing format, such as margins, headers, and footers.

The Page Setup window consists of several tabs. Use the OK button to save changes and exit the Page Setup window. Click the Cancel button to exit the Page Setup window without saving changes. Click the Apply button to update the parameters without closing the Page Setup window.

Note: On each tab in the Page Setup window, certain items are checked (selected) by default. To deselect these options, click the item.

Data Tab

The Data tab displays a list of items that can be selected for printing. Select the desired items and click the OK button.

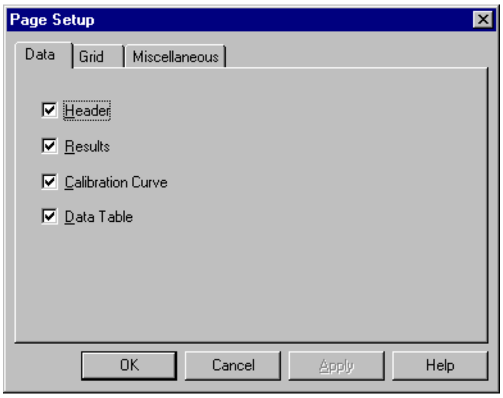


Figure A.9 Page Setup Window: Data Tab

Grid Tab

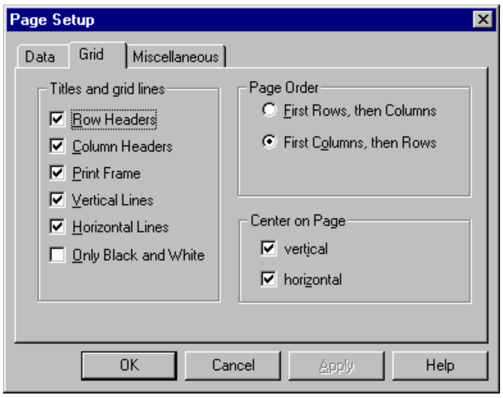


Figure A.10 Page Setup Window: Grid Tab

The Grid tab displays a list of format items for the report, as described below.

Option	Description
Row Headers	Prints row headers in the table.
Column Headers	Prints column headers in the table.
Print Frame	Prints a frame around the table.
Vertical Lines	Prints vertical lines in the table.
Horizontal Lines	Prints horizontal lines in the table.
Only Black and White	Prints only in 2 colors, without gray backgrounds or other colors.
First Rows, then Columns	Prints rows first, followed by columns, when more than one page is printed
First Columns, then Rows	Prints columns first, followed by rows, when more than one page is printed.
Vertical	Prints the table on the vertical center of the page.
Horizontal	Prints the table on the horizontal center of the page.

Miscellaneous Tab

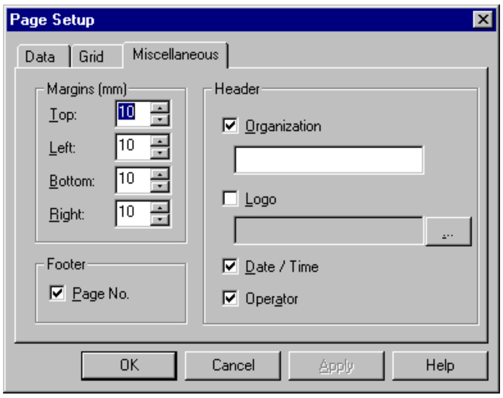


Figure A.11 Page Setup Window: Miscellaneous Tab

The Miscellaneous tab displays a list of format items for the report, as described below.

Setting Item	Description
Margins	Use the spin controls to enter the page margins (in mm).
Footer, Page No.	Select this option to print the page number in the footer of each page.
Header	*Organization*: Select this option and enter the name of the organization in the text box to print the organization name in the page header. *Logo*: Select this option to print a bitmap in the page header. Use the Browse button to select the bitmap file. *Date/Time*: Select this option to print the current system date and time in the page header. *Operator:* Select this option to print the current user name in the page header.

A.2.9 Exit

Select Exit to close the Method Validation application. If an active file contains unsaved information, a prompt to save changes is displayed. Select Yes or No, or select Cancel to abort the exit function.

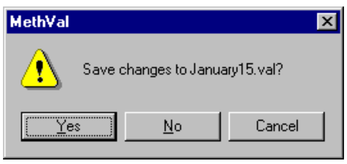


Figure A.12 Prompt to Save Data Changes

A.3 Edit Menu

Options such as Cut, Copy, and Paste are available only if content is selected.

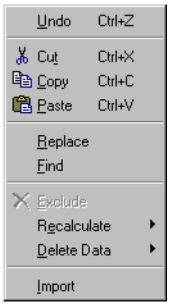


Figure A.13 Edit Menu

A.3.1 Undo

Use the Undo function to reverse the last change made in the data table. This function is available only in documents that can be manually edited and when the user changes the content of a cell in the data table.

A.3.2 Cut/Copy/Paste

Use the Cut, Copy, and Paste commands to edit highlighted table cells and rows. The Cut command deletes information in rows or cells and stores it on the clipboard. The Copy command copies data from rows or cells to the clipboard. The Paste command copies the contents of the clipboard to the current row of the table. When pasting a whole row, select the entire destination row first by clicking the row number.

Note: Do not select a row for pasting that currently contains data. The data will be overwritten!

TIP: *The Cut, Copy and Paste commands have shortcut buttons on the toolbar.*

A.3.3 Recalculate

Use the Recalculate command to recalculate all method validation data. The results and calibration curve sections of the Method Validation window are also updated when the Recalculate command is selected. Errors that occur during the recalculation process are displayed in message boxes. For example, an error message may be displayed if the data in the table does not contain sufficient information, if there are not enough data points defined in the table, if less than 3 injections are available, and if numeric data are incorrect and results in an illegal mathematical operation (such as division by zero).

TIP: *The Recalculate command has a shortcut button on the toolbar.*

A.4 View Menu

Figure A.14 View Menu

Toolbar

The Toolbar function displays or hides the toolbar.

Status Bar

The Status Bar function displays or hides the status bar.

A.5 Options Menu

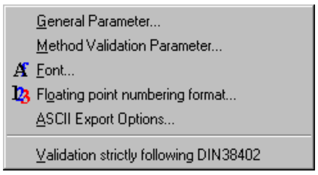


Figure A.15 Options Menu

A.5.1 General Parameter

Select the General Parameter command to display the General Information dialog box.

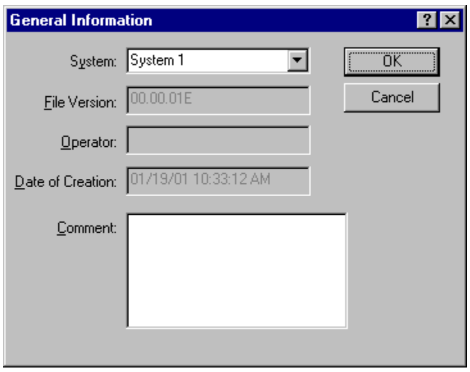


Figure A.16 General Information Dialog Box

The General Information dialog box is used to view and edit information about the method validation file, such as the system used to create the file, the file version, the operator who created the file and the date on which the file was created. Some information fields may be disabled. Comments about the file are displayed in the Comment field.

A.5.2 Method Validation Parameter Window

The Method Validation Parameter command displays the Method Validation Parameter window, which contains detailed information about the method, as described below.

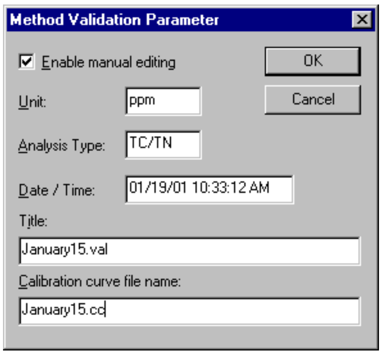


Figure A.17 Method Validation Parameter Window

Item	Description
Enable manual editing	Select to allow data tables to be edited manually. This option is disabled for files linked to TOC-Control V calibration curves. If this option is deselected, it is disabled and cannot be selected again. This option is selected by default.
Unit	Displays the calibration curve units. This option is disabled if the Enable manual editing option is not selected.
Analysis Type	Displays the analysis used to create the calibration curve. This option is disabled if the Enable manual editing option is not selected.
Date/Time	Displays the date and time the file was created.
Title	Displays the title of the method validation file.
Calibration curve file name	Displays the name of the TOC-Control V calibration curve.

A.5.3 Font

Use the Font command to select fonts for printed reports and display screens. Selecting the Font command opens the Font dialog box.

TIP: *The Font command has a shortcut button on the toolbar.*

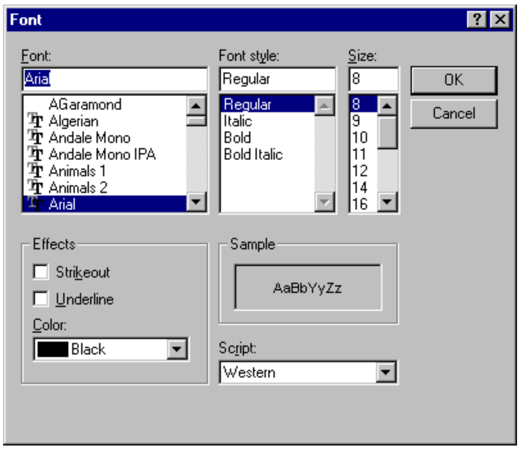


Figure A.18 Font Dialog Box

Choose a font for printed reports by selecting options from the Font, Font Style, Size, Color and Script drop-down lists. A preview of the font appearance is visible in the Sample field on the lower portion of the Font dialog box.

A.5.4 Floating Point Numbering Format

Use the Floating Point Numbering Format command to select display options for numeric results in the method validation tables. These options apply only to the manner in which numbers are displayed; internal data retain their original values.

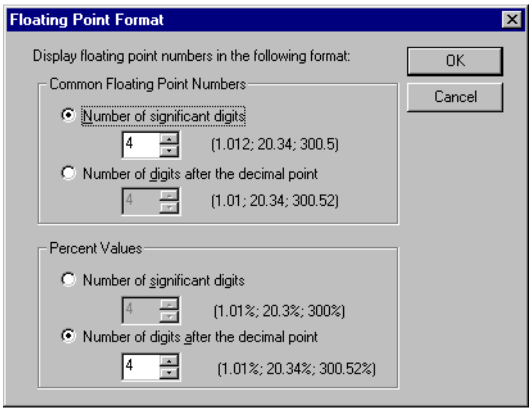


Figure A.19 Floating Point Format Window

Common Floating Point Numbers

Setting Item	Description
Number of significant digits	Select this option to display common floating point numbers using a specified number of significant digits. Use the spin control to set the number of significant digits. The default value is 4.
Number of digits after the decimal point	Select this option to display common floating point numbers using a specified number of digits after the decimal point. Use the spin control to set the number of digits after the decimal point. The default value is 2.

Percent Values

Setting Item	Description
Number of significant digits	Select this option to display percent values using a specified number of significant digits. Use the spin control to set the number of significant digits. The default value is 4.
Number of digits after the decimal point	Select this option to display percent values using a specified number of digits after the decimal point. Use the spin control to set the number of digits after the decimal point. The default value is 2.

A.5.5 ASCII Export Options

Use the ASCII Export Options function to export method validation data to an ASCII file.

Figure A.20 ASCII Export Options Dialog Box

Setting Item	Description
Header	Select to export method validation file header.
Data	Select to export method validation data.
Results	Select to export method validation results.
Separator	Select the type of separator to be used between items in the ASCII file.
Export strings in quotation marks	Select this option to add quotation marks to all exported strings.

To export a file, create or open a method validation file. Select the desired options from the ASCII Export Options dialog box and select OK. The File>Save As dialog box is displayed. Enter a file name for the ASCII file and select the *.txt file type.

A.5.6 Validation Strictly Following DIN38402

Use the Validation Strictly Following DIN38402 option to conduct the validation calculation by DIN 38402 requirements; that is, only the area obtained from the first injection of every standard is used in the calibration calculation. When this option is not selected, the mean value of the area counts from all injections of each standard is used in the calibration calculation.

Perform method validation according to DIN regulations according to the following steps:

1. Select the estimated working range.

2. Prepare 10 calibration standards, with concentrations distributed uniformly over the complete working range.

3. Create a Sample Table, with 10 injections of the highest and lowest standard concentrations and a single injection for all other standards. If more than one injection is made of the intermediate standards, the linearity test is calculated with the results of the first replicates only.

4. Analyze the standards. The results are saved in a.cal file.

5. Select Method Validation from the Tools menu in the Sample Table Editor. The Method Validation main window is displayed.

6. Select the Validation Strictly Following DIN from the Options menu.

7. Select Open from the File menu.

8. Select.cal from the file type drop-down list.

9. From the list of file names, select the.cal file where the measurement data was saved. Then select OK.

10. The calculations are automatically performed, and the validation data displayed.

11. Select Save from the File menu, assign a file name in the dialog box, and select OK. The validation data are saved with a.val file name extension.

When the Validation Strictly Following DIN option is enabled, a check mark is displayed in front of the menu item. After selecting this option, a confirmation message is displayed.

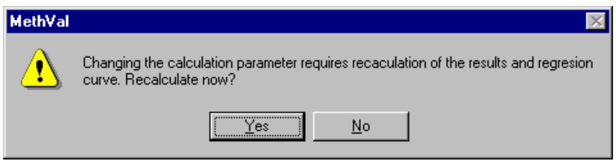


Figure A.21 Confirmation Message for Validation Strictly Following DIN 38402

Select Yes to execute recalculation. The calibration curve and result sections of the Method Validation window are updated accordingly.

A.6 Help Menu

About Meth Val...

Use the About command to view copyright and version information.

Appendix B

Control Charts

The Control Charts software monitors precision and accuracy of a defined data set. The software supports five types of Control Charts:

•Mean Value Control Charts

•Recovery Control Charts

•Blind Value Control Charts

•Spanwidth Control Charts

•User Defined Control Charts

The Mean Value Control Chart checks the accuracy of an analytical method on an instrument. Control value drifts may indicate a change in system performance.

The Recovery Control Chart tests an analytical method for matrix influences and provides a restrictive control of accuracy by testing proportional system errors. Using certified standards will test the accuracy of the system as well as the recovery rate.

The Blind Value Control Chart is a special form of the Mean Value Control Chart used for quality assurance of the instrument and variable elements. Unlike the Mean Value Control Chart, the control value is the information value (absorbance, peak height, peak area) received from the analysis.

The Spanwidth Control Chart tests the precision of an analytical method and monitors the accuracy under actual conditions (Drift Control). For this type of Control Chart, there are several existing models for calculating the control and warning limits.

Each type of control chart can be used to create charts for different working range and analysis methods. For each control chart, a file is created which contains general information about the type, data structure and other global data.

B.1 File Menu

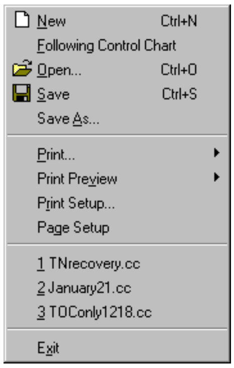


Figure B.1 File Menu

B.1.1 New

The New function is used to define new control charts and displays the Control Chart Wizard. Follow the Control Chart Wizard process to enter a new control chart. The file extension *.cc is used for all control charts.

Note: Control Chart parameters can be changed at a later time by selecting Options>Control chart options from the main menu bar.

Control Chart Wizard System Information Window

The first page of the Control Chart Wizard is used to enter basic system information and is similar to the System Information pages in the Method and Sample Wizards Refer to Section 4.4.5.2 “Sample” and Section 4.4.2.3 “Method Wizard” for more details.

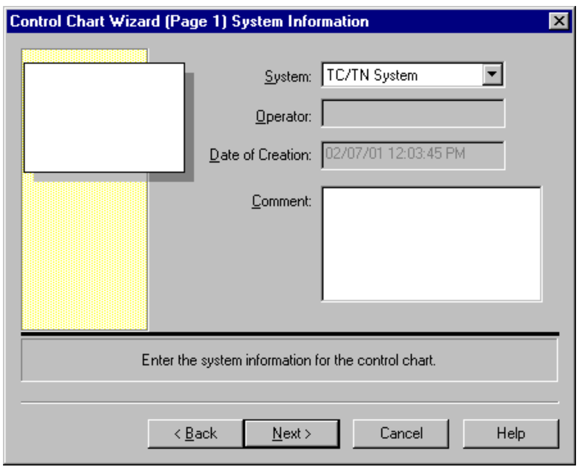


Figure B.2 Control Chart Wizard: System Information Window

Control Chart Wizard Control Chart Type Window

Select the type of control chart to be created and select the analysis type from the drop down list.

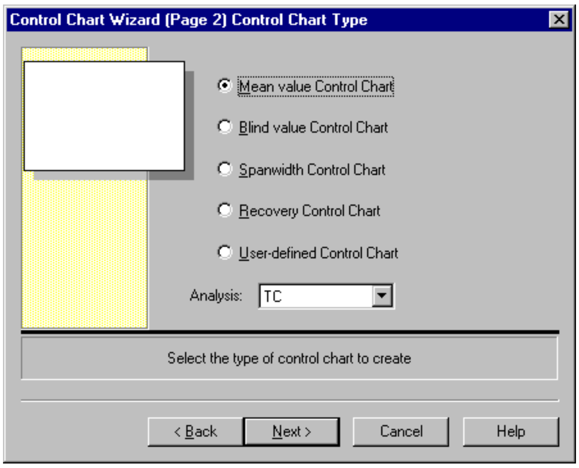


Figure B.3 Control Chart Wizard: Control Chart Type Window

Control Chart Wizard Control Chart Parameters Window

Enter the control chart parameter values and limits as described below.

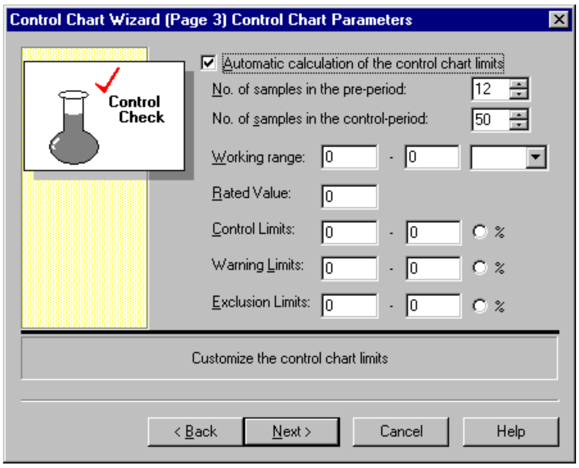


Figure B.4 Control Chart Wizard: Control Chart Parameters Window

Setting Item	Description
Automatic calculation of the control chart limits	Select to enable automatic calculation of the control chart limits. The upper and lower warning and control limits are calculated after the control samples for the preparation period are added to the control chart.
No. of samples in the pre-period	Enter the number of samples in the preparation period. The default value is 12. This option is disabled if the Automatic calculation of the control chart limits option is not selected.
No. of samples in the control-period	Enter the number of samples in the control period. The default value is 50.
Working range	Enter the upper and lower limits of the working range. Match the range with the concentration of samples used for control tracking and the concentration range of the selected calibration curve. Use the drop-down list to select the units.
Rated Value	Enter the rated value of the working range. This option is disabled if the Automatic calculation of the control chart limits option is not selected.
Control Limits	Enter the upper and lower control limits. This option is disabled if the Automatic calculation of the control chart limits option is not selected.
Warning Limits	Enter the upper and lower warning limits. This option is disabled if the Automatic calculation of the control chart limits option is not selected.
Exclusion Limits	Enter the upper and lower exclusion limits.

Control Chart Wizard Recovery Control Chart Parameter Window

This window is only displayed if the Recovery Control Chart is selected in the Control Chart Wizard, and Automatic calculation of the control chart limits is not selected.

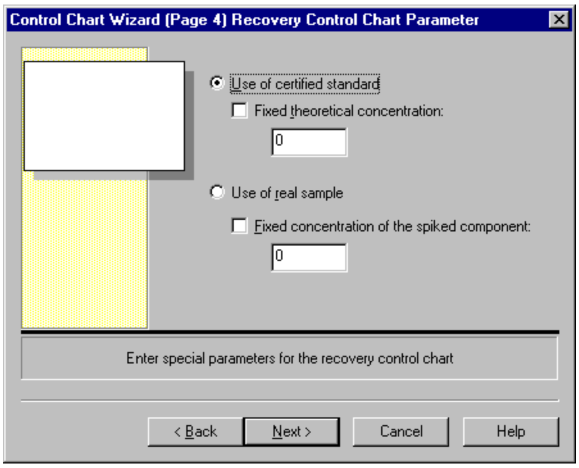


Figure B.5 Control Chart Wizard: Recovery Control Chart Parameter Window

Setting Item	Description
Use of certified standard	Select this option to use a certified sample as a control sample.
Fixed theoretical concentration	Select this option if the standard has a fixed concentration. Enter the concentration in the text box.
Use of real sample	Select this option if the control sample is a real sample.
Fixed concentration of the spiked component	Select this option if the spiked component has a fixed concentration. Enter the concentration in the text box.

Control Chart Wizard Spanwidth Control Chart Options Window

This window is only displayed if the Spanwidth Control Chart is selected in the Control Chart Wizard.

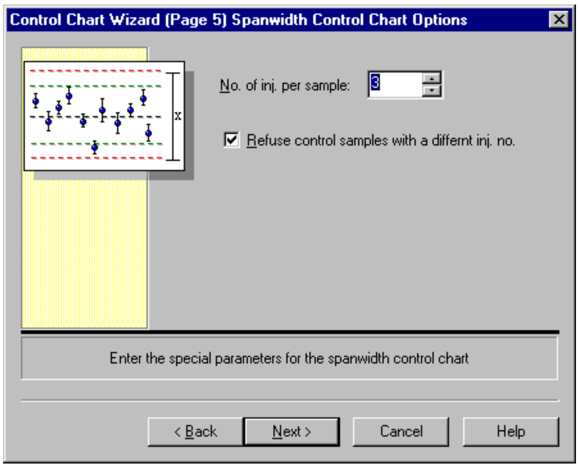


Figure B.6 Control Chart Wizard: Spanwidth Control Chart Options Window

Setting Item	Description
No. of inj. per sample	Enter the number of injections for a sample used in the control chart. The default value is 3, and the range is 3-25.
Refuse control samples with different inj. no	Select this option to exclude from the control chart samples whose number of injections differs from those specified in the No. of inj. per sample field.

Control Chart Wizard History Window

The Control Chart Wizard History function is identical to other history functions in the TOC-Control V system. Refer to Section 4.4.2.3 “Method Wizard” for more details about the History function. The History window provides information on all control chart changes when enabled.

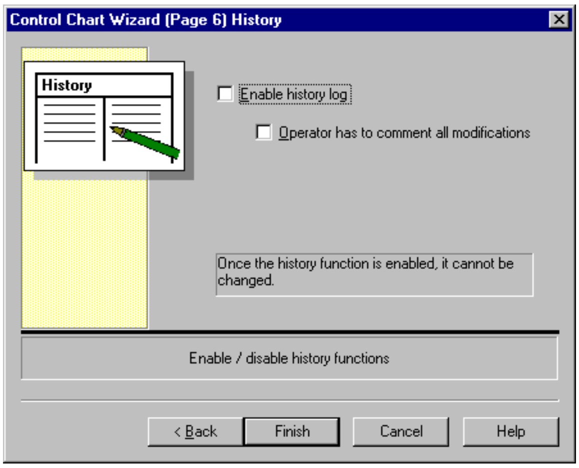


Figure B.7 Control Chart Wizard: History Window

Setting Item	Description
Enable history log	Select this option to enable the History Log, which is used to monitor all modifications to the control charts. Once the History Log is enabled, it cannot be turned off.
Operator has to comment all modifications	Select this option to require the user to enter comments or obtain approval for modifications to the control charts.

B.1.2 Following Control Chart

Select the Following Control Chart menu item to create a Following Control Chart. It is recommended that a new control chart be created after 50 samples have been entered in the control period. The new control chart uses the calculated standard deviation from the control period as the new control chart limits. Control chart options are:

Setting Item	Description
File Name	Displays the name of the currently active control chart.
Save the current control chart as	Enter a new file name in this field to save the currently active control chart to a new location or with a new file name. A confirmation message is displayed to confirm the operation.
Create	Select this option to create the Following Control Chart. The new limits are calculated from the samples of the control period stored in the previous control chart.
Parameter	Select this option to display the parameters of the newly created Following Control Chart. This function can also be accessed using the Options>Control Chart Options command in the main window.

B.1.3 Open

The Open command is used to open files. Select a *.cc file to open a control chart file. Refer toSection 4.4.2.4 "Open" for further information on the standard File>Open dialog box.

B.1.4 Save

The Save command saves the current control chart.

B.1.5 Save As

Use the Save As command to save files for the first time, or to save a file using a new file name. Files can be saved as file types *.cc or *.txt (ASCII format). Refer to Section 4.4.2.7 "Save As" for more information on the Save As function.

B.1.6 Print>Data

Use the Print>Data command to print data in spreadsheet format. Refer to Section 4.4.2.10 “Print>Table” for more information on the Print function.

B.1.7 Print>Graph

Use the Print>Graph command to print data in graphical format. Refer to Section 4.4.2.10 “Print>Table” for more information on the Print function.

B.1.8 Print Preview>Data/Print Preview>Graph

Use the Print Preview>Data function to display a preview of the data to be printed. Use the Print Preview>Graph function to display a preview of the graph to be printed. Refer to Section 4.4.2.12 “Print Preview>Table” for more information on the Print Preview function.

B.1.9 Print Setup

Use the Print Setup command to select the printing parameters.

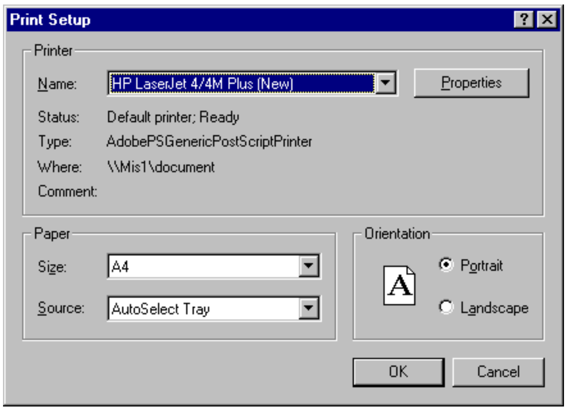


Figure B.8 Print Setup Dialog Box

The print options available vary based on the printer installed. Select the paper size and source from the drop-down lists, and select Portrait or Landscape to change the orientation of the printed file. Select OK to save the selections, or Cancel to close the dialog box without saving changes.

B.1.10 Page Setup

Use the Page Setup command to select items to be printed and to select options for printing format, such as margins, headers, and footers.

The Page Setup window consists of several tabs. Use the OK button to save changes and exit the Page Setup window. Click the Cancel button to exit the Page Setup window without saving changes. Click the Apply button to update the parameters without closing the Page Setup window.

Note: On each tab in the Page Setup window, certain items are checked (selected) by default. To deselect these options, click the item.

Data Tab

The Data tab displays a list of items that can be selected for printing. Select the desired items and click the OK button.

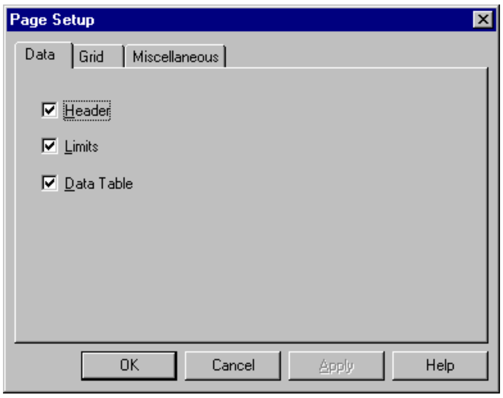


Figure B.9 Page Setup Window: Data Tab

Grid Tab

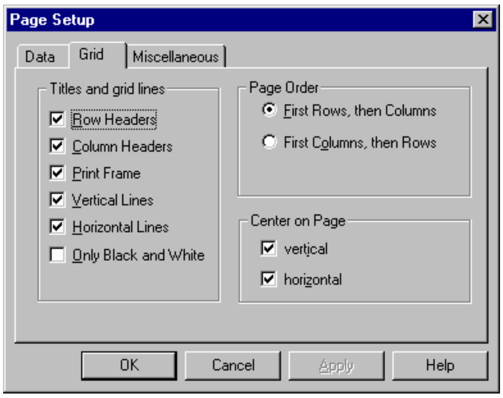


Figure B.10 Page Setup Window: Grid Tab

The Grid tab displays a list of format items for the report, as described below.

Option	Description
Row Headers	Prints row headers in the table.
Column Headers	Prints column headers in the table.
Print Frame	Prints a frame around the table.
Vertical Lines	Prints vertical lines in the table.
Horizontal Lines	Prints horizontal lines in the table.
Only Black and White	Prints only in 2 colors, without gray backgrounds or other colors.
First Rows, then Columns	Prints rows first, followed by columns, when more than one page is printed
First Columns, then Rows	Prints columns first, followed by rows, when more than one page is printed.
Vertical	Prints the table on the vertical center of the page.
Horizontal	Prints the table on the horizontal center of the page.

Miscellaneous Tab

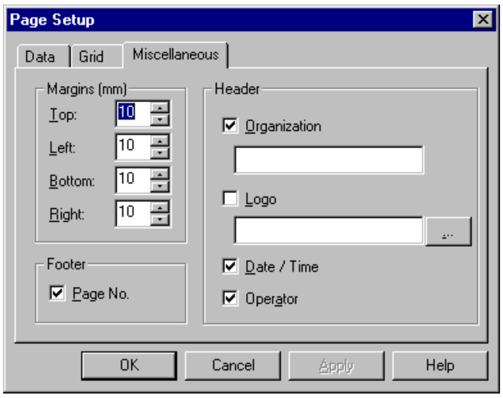


Figure B.11 Page Setup Window: Miscellaneous Tab

The Miscellaneous tab displays a list of format items for the report, as described below.

Setting Item	Description
Margins	Use the spin controls to enter the page margins (in mm).
Footer, Page No.	Select this option to print the page number in the footer of each page.
Header	Organization: Select this option and enter the name of the organization in the text box to print the organization name in the page header. Logo: Select this option to print a bitmap in the page header. Use the Browse button to select the bitmap file. Date/Time: Select this option to print the current system date and time in the page header. Operator: Select this option to print the current user name in the page header.

B.1.11 Exit

Select Exit to close the Control Chart application.

B.2 Edit Menu

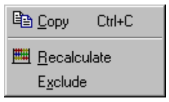


Figure B.12 Edit Menu

Copy

Use the Copy command to copy the selected range from the data table to the clipboard.

Recalculate

Use the Recalculate command to recalculate the limits of the control chart. This function should be performed after one or more control samples have been excluded or added to the control chart.

Exclude

Use the Exclude command to exclude a selected range of control samples from the control chart calculations. When Exclude is selected, the Exclude dialog box is displayed. Enter a comment describing the reason for excluding the sample. The Exclude function is only available when one or more control samples are selected. A dialog box displays a message requesting confirmation that the sample be excluded. Select Yes to confirm and recalculate the control limits, or No to abort recalculation.

B.3 View Menu

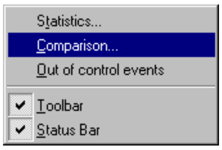


Figure B.13 View Menu

B.3.1 Statistics

The Statistics command displays the Statistics window.

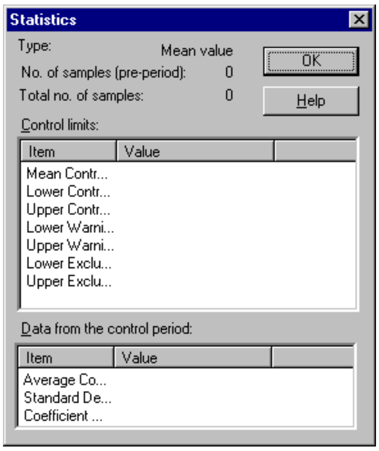


Figure B.14 Statistics Window

Setting Item	Description
Type	Displays the type of control chart.
No. of samples (pre-period)	Displays the number of samples in the preparation period. This value was entered in the Control Chart Wizard.
Total no. of samples	Displays the total number of samples in the control chart. Excluded samples are not included in the total number.
Control limits	Displays the control limits.
Data from the control period	Displays the Average Control Value, which is the mean value of the samples in the control period. Also displays the calculated standard deviation and coefficient of variation.

B.3.2 Comparison

After a control chart is complete, the results from the control period must be compared with the results from the preparation period. For control charts without a preparation period, the current values must be compared with the results of the preparation period or the control period from the previous control chart. The View>Comparison command displays the Comparison between control charts window.

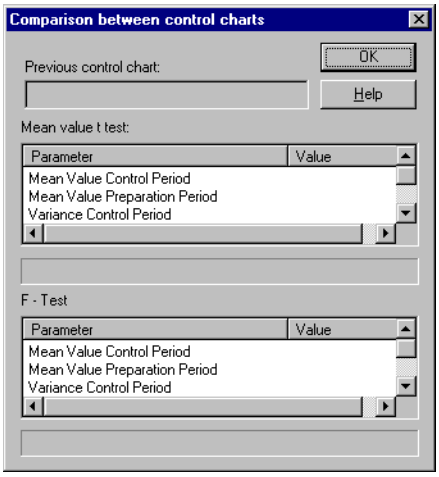


Figure B.15 Comparison Between Control Charts Window

Setting Item	Description
Previous control chart	Displays the file name of the previous control chart, if applicable.
Mean value t-test	Displays the results of the T-test in the text box beneath the Item and Value lists. The T-table value and the calculated test value are also displayed in the Item and Value list. The test value is calculated based on the mean values and variances obtained from the control and preparation period. The T-test result shows whether the long term deviation of the control samples is significant.
F-test	Displays the results of the F-test in the text box beneath the Item and Value list. The F-table value and the calculated test value are also displayed in the Item and Value list.

B.3.3 Out of Control Events

The Out of Control Events window displays a list of all out of control events.

Figure B.16 Out of Control Events Window

Spl. No.

Displays the sample number for which the out of control event occurred.

Event

Describes the out of control event.

B.3.4 Toolbar/Status Bar

The View>Toolbar and View>Status Bar commands show and hide the Toolbar and Status Bar, respectively.

B.4 Options Menu

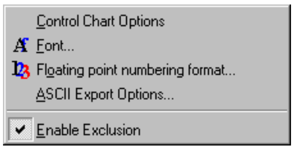


Figure B.17 Options Menu

B.4.1 Control Chart Options

Use the Control Chart Options command to view and edit the control chart parameters entered in the various pages of the Control Chart Wizard. Refer to Section B.1.1 “New” for more details.

Note: Some options within the Control Chart Options tabs (such as control chart type or sample concentrations) may be disabled if the control chart contains data. The disabled parameters are options that cannot be modified once data are entered.

B.4.2 Font

The Font command opens the standard Font dialog box. Refer to Section 4.4.9.3 “Display Settings>Display Font” for detailed information about font options.

B.4.3 Floating Point Numbering Format

The Floating Point Numbering Format command is used to select display options for numeric results in the control charts. These options apply only to the manner in which numbers are displayed; internal data retain their original values.

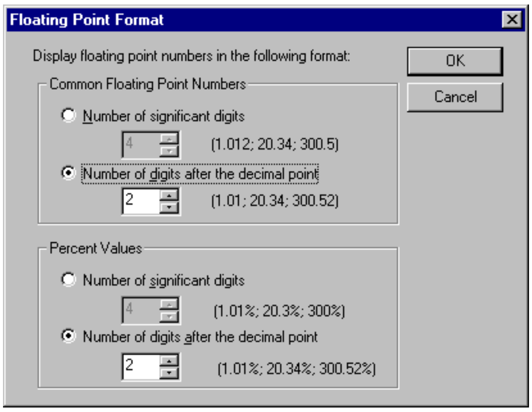


Figure B.18 Floating Point Format Window

Common Floating Point Numbers

Setting Item	Description
Number of significant digits	Select this option to display common floating point numbers using a specified number of significant digits. Use the spin control to set the number of significant digits. The default value is 4.
Number of digits after the decimal point	Select this option to display common floating point numbers using a specified number of digits after the decimal point. Use the spin control to set the number of digits after the decimal point. The default value is 2.

Percent Values

Setting Item	Description
Number of significant digits	Select this option to display percent values using a specified number of significant digits. Use the spin control to set the number of significant digits. The default value is 4.
Number of digits after the decimal point	Select this option to display percent values using a specified number of digits after the decimal point. Use the spin control to set the number of digits after the decimal point. The default value is 2.

B.4.4 ASCII Export Options

The ASCII Export Options function is used to export control chart data to an ASCII-formatted file.

Figure B.19 ASCII Export Options Dialog Box

Setting Item	Description
Header	Select to export control chart file header.
Data	Select to export control chart data.
Limits	Select to export control chart limits.
Separator	Select the type of separator to be used between items in the ASCII file.
Export strings in quotation marks	Select this option to add quotation marks to all exported strings.

To export a file, create or open a control chart. Select the desired options from the ASCII Export Options dialog box, and select OK. The File>Save As dialog box is displayed. Enter a file name for the ASCII file and select the *.txt file type.

B.4.5 Enable Exclusion

Use the Enable Exclusion command to exclude a selected range of control samples from the control chart calculations. When the Enable Exclusion command is executed, the Exclude dialog box is displayed. Enter a comment describing the reason for excluding the sample.This option is only available when one or more control samples are selected. A dialog box displays a message requesting confirmation that the sample be excluded. Select Yes to confirm and recalculate the control limits, or No to abort the process.

B.5 Help Menu

About ContrCharts...

Use the About command to view copyright and version information.

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