638-94249A (M393-E198)

Solid Sample Module

SSM-5000A

for TOC-V Series Total Organic Carbon Analyzers

User Manual

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

ANALYTICAL & MEASURING INSTRUMENTS DIVISION KYOTO, JAPAN

Solid Sample Module

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Introduction

Thank you for purchasing the SSM-5000A Solid Sample Module for the Shimadzu TOC-V series Total Organic Carbon Analyzer.

Be sure to read this manual before using the SSM-5000A accessory. Keep the manual close at hand for future reference.



- Do not use the SSM-5000A until you fully understand the contents of this manual.
- If this manual or the warning labels become lost or damaged, promptly obtain replacements from your Shimadzu representative.
- To ensure safe operation of this unit, read and follow the Operational Precautions before operating the instrument.

Product Warranty and Post Sale Service

Product Warranty

The warranty does not cover malfunctions that result from:

- incorrect operation;
- repairs or modifications not conducted by Shimadzu or a designated representative;
- external factors;
- operation under extreme environmental conditions, such as high temperature and humidity, corrosive gas, or excessive vibration;
- fire, earthquakes, or other natural disasters;
- moving or transporting the instrument after the initial installation;
- consumption of items or parts that can be regarded as consumable.

Post Sale Service

If the instrument is not working properly, follow the measures described in Section "4.2 Troubleshooting". Contact your Shimadzu representative if the malfunction cannot be remedied or if problems arise that are not listed in the Troubleshooting section of this manual.

Contents of this Manual

1 - Overview

This section gives an overview of the instrument and describes precautions to ensure its safe use.

2 - System Components

This section describes the names and major functions of the various components of the SSM-5000A.

3 - Operation

This section describes screen displays and procedures for pre-analysis preparation of the instrument, sample analysis, and calibration curve measurement.

4 - Maintenance

This section describes maintenance, servicing and other important procedures for operating the instrument. Error messages and troubleshooting procedures are also described.

5 - Reference Information

This section describes the instrument specifications, accessories, and consumables. The appropriate installation conditions are explained. Refer to this section when moving the instrument.

Notation Conventions

Precaution Conventions Used in this Manual

The following precautionary conventions are used in this manual.

Convention	Description	
CAUTION	Failure to avoid this problem may lead to light or medium injury or may result in physical damage.	

Note: Information to correctly use the instrument.

Other Conventions Used in this Manual

In addition to precautionary conventions, tips and references are also used to supply additional information.

Convention

Description

 $\underline{\text{TIP }} \text{$ **w** $} Handy hints, alternative methods, or other useful advice.}$

<u>Reference:</u> Where to find detailed information.

Warning Labels

High Temperature Warnings

When the electric furnace is heating or has reached the operating temperature (900°C for TC and 200°C for IC), the internal parts are at high temperatures. Due to risk of burn injury, wait until the furnace cools to room temperature before opening the cover. Only perform maintenance to the combustion tube or catalyst after they have returned to room temperature. The furnace temperature is displayed in the SSM-5000A temperature display window.



The area around the electric furnaces becomes extremely hot during furnace heating or when the furnaces reach their operating temperatures (TC furnace: 900°C, IC furnace: 200°C). Because of the danger of burn injuries, never touch these areas with bare hands. Allow the furnaces to cool before conducting maintenance on the combustion tube or catalyst.



Corrosion Warning

The drain liquid may contain acid or other corrosive substances. Use care to avoid touching or spilling the drain liquid.



Phosphoric acid is strongly acidic. Use care to avoid touching or spilling the phosphoric acid.



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This chapter gives an overview of the instrument and describes the operational precautions for safe instrument use.

1.1 Overview

This section provides an overview of the instrument.

1.2 Operational Precautions This section describes precautions to ensure that the instrument is used correctly.

1.1 Overview

The SSM-5000A is a special accessory for the TOC-V series Total Organic Carbon Analyzer which combines with the TOC-V to create a TOC Solid Sample Analysis System capable of analyzing solid samples, such as soil.

This manual describes the operation of the SSM-5000A Solid Sample Module alone and also the operation of the TOC-V with the SSM-5000A attached. For other procedures related to the TOC-V, refer to the appropriate User Manual.

To ensure that the SSM-5000A is used correctly, be sure to read this User Manual before using the SSM-5000A. The manual should be conveniently stored for future reference.



Safety may be compromised if the instrument is used in a fashion other than indicated in this User Manual. Pay particular attention to the CAUTION sections to ensure safe operation of the instrument.

1.2 Operational Precautions

Strictly follow the precautions below when using the SSM-5000A.



- When the electric furnace is heating or when it has reached operating temperature, the internal parts are at high temperature. Due to risk of burn injury, be sure to wait until the furnace oven cools to room temperature before opening the cover to perform maintenance related to the combustion tube or catalyst.
- The SSM-5000A is capable of measuring solids, suspensions, and solutions. The solid sample can have many attributes that affect the result, such as shape, matrix, and water content. The efficiency of the carbon combustion oxidation reaction (TC analysis) and carbonate acidification reaction (IC analysis) may vary with the sample type. The TC or IC recovery rate of some samples may be low. IC analysis of some samples may be difficult due to insufficient mixing with the phosphoric acid added for acidification, or because of solidification due to a reaction with the phosphoric acid. Determine the optimal parameters before starting sample analysis. Particle size, dryness, sample weight, carrier gas flow rate, whether the sample boat is covered, selection of additives, and types of standards used for calibration are examples of the parameters that need to be predetermined.
- If a sample contains highly volatile TOC components, before measuring place the sample boat in the sample port then cover it immediately. This precaution may not entirely prevent a low bias due to loss of volatile components during sampling, weighing, or analysis.
- Never analyze explosive samples. Explosions could injure people nearby and damage the instrument.
- Never analyze samples that emit toxic substances during combustion, such as samples containing mercury or arsenic.
- Do not directly analyze seawater or water with a salt content equivalent to seawater. Dilute these samples to lower the salt concentration before analysis. If analyzed directly, substances emitted during TC combustion or IC reaction may hinder the analysis or damage the interior of the detector.
- When using the TOC Solid Sample Analysis System, carrier gas must flow through both the 200mm and 1.0mm cells of the tandem NDIR. Even when a liquid sample is analyzed using the TOC-V only, the SSM-5000A carrier gas must flow through the 1.0mm cell as purge gas. To minimize the SSM-5000A carrier gas consumption, the gas flow rate may be decreased to 50mL/min.
- Since the gas flow lines of the TC and IC sample ports are connected in series, ensure that both ports are fully closed during analysis.
- Clean the instrument with a damp cloth; do not use chemicals.
- Avoid injury by contacting your Shimadzu representative to perform any repairs to the interior of the instrument.

- Never disassemble or modify the instrument beyond the range of maintenance described in the Instruction Manual. Safety may be compromised.
- This instrument does not have a flameproof construction. Do not use it in a dangerous area.
- Utilize a 100 to 115VAC power supply (100V series) or a 220 to 240VAC power supply (200V series).

System Components

2

This chapter describes the names and major functions of the various components of the SSM-5000A.

- 2.1 *Front View* Diagram of the front of the instrument with the name and functions of each part.
- 2.2 *Top View* Diagram of the top of the instrument with the name and functions of each part.
- 2.3 Right View

Diagram of the right side of the instrument with the name and functions of each part.

2.4 Rear View

Diagram of the rear of the instrument with the name and functions of each part.

2.5 Flow Diagrams

Flow diagrams of the SSM-5000A alone and in combination with the TOC-V main instrument.

2.1 Front View



Figure 2.1 • SSM-5000A Front View

No.	Item	Description
1	Display window	Displays the TC furnace temperature, IC furnace tempera- ture, and power supply ON/OFF status.
2	Sample port cover (IC side)	IC sample port cover. This green cover can be moved up, down, left, and right.
3	Sample port cover (TC side)	TC sample port cover. This blue cover can be moved up, down, left, and right.
4	Sample boat push rod knob (IC side)	Use this green knob to move the sample boat in and out of the IC furnace.
5	Sample boat push rod knob (TC side)	Use this blue knob to move the sample boat in and out of the TC furnace.
6	Sample port (IC side)	The sample boat is inserted and removed through the open- ing at the top of the IC port.
7	Sample port (TC side)	The sample boat is inserted and removed through the open- ing at the top of the TC port.
8	Sample port cover knob (IC side)	Turn this green knob counterclockwise to raise the IC sam- ple port cover. Move the knob to the right to open the sam- ple port.
9	Sample port cover knob (TC side)	Turn this blue knob counterclockwise to raise the TC sam- ple port cover. Move the knob to the right to open the sam- ple port.
10	Flow controller	Controls the carrier gas flow. See "Figure 5.4 • Flow Path of the SSM-5000A Flow Controller" and "Figure 3.7 • Setting the SSM-5000A Carrier Gas Flow Rate"

2.2 Top View



Figure 2.2 • Top View

No.	Item	Description
1	Sample boat stop position indicators	Placement indicators used to move the sample boat into and out of the furnace.
2	IC reaction tube	Made of Pyrex [®] glass.
3	TC combustion tube	Made of quartz glass and filled with an oxidizing catalyst.
4	IC furnace	Normal operating temperature is 200°C.
5	TC furnace	Normal operating temperature is 900°C.
6	Condensation coil	Cools the gases generated by sample combustion in the TC combustion tube.

2.3 Right View



Figure 2.3 • SSM-5000A Right View

No.	Item	Description
1	Power switch	Used to turn the power to the unit ON/OFF.
2	Drain vessel	Water separated by the drain separators $\textcircled{0}$ enters this drain vessel. Water that overflows the drain vessel is drained through tubing $\textcircled{3}$ to the outside of the unit.
3	Drain tubing	Water that overflows the drain vessel exits the instrument through this tubing.
4	Drain separators	Independently attached to the TC and IC flow paths to con- dense and collect any water generated from the carrier gas in the TC combustion tube or IC reaction tube.
5	Carrier gas flow stabilizer	Stabilizes the flow of carrier gas to ensure that CO_2 generated during sample combustion is sent to the NDIR.
6	Sample boat push rod	Used to move the sample boat into the TC furnace or IC furnace.
7	Sample boat holder	Supports the sample boat when it is moved.
8	Sample boat	Made of alumina ceramic.
9	O-ring adapter	Holds the O-ring in place to form the seal where the sample boat push rod enters the sample port block inlet.

2.4 Rear View



Figure 2.4 • SSM-5000A Rear View

No.	Description	Major functions
1	Carrier gas inlet	Connector for the SSM-5000A carrier gas (oxygen).
2	Carrier gas outlet	Carrier gas outlet tube protrudes from this outlet to expel the carrier gas and the CO_2 generated in the TC combustion tube and/or the IC reaction tube.
3	Power socket	Connector for the 100VAC \pm 10% power supply.
4	Driver board connector	Connect the TOC-V digital I/O port board to this connector for signal transmission between the TOC-V and the SSM-5000A.
5	Rear panel	Provides access to the rear of the instrument.
6	Drain nipple	Outlet for drain water that overflows from the drain vessel.
7	Cooling coil	Condenses water vapor from the carrier gas using ambient air to cool the hot carrier gas from the TC combustion tube and IC reaction tube.

2.5 Flow Diagram

2.5.1 SSM-5000A Flow Diagram



Figure 2.5 • SSM-5000A Flow Diagram

2.5.2 TOC-V + SSM-5000A Flow Diagram



Gas is supplied to the TOC-V and SSM-5000A from a single high-purity oxygen cylinder.

Figure 2.6 • TOC-V & SSM_5000A Flow Diagram

2.5 Flow Diagram2.5.2 TOC-V + SSM-5000A Flow Diagram



This chapter describes the analysis preparations, parameter entry procedures and operation procedures for calibration curve generation and sample analysis.

3.1 Analysis Preparations

This section describes solid sample pulverizing, drying and other sample preparation procedures; inserting sample into the sample boat; preparing SSM-5000A standards, creating calibration curves, and preparations for analyzing solid samples.

3.2 Starting Up the Instrument

This section describes the procedure for starting up the instrument.

3.3 Measurement Using the TOC-VCSH/CSN/WS

This section describes procedures for setting analytical parameters, performing calibration curve and sample measurement and reviewing data. Detailed descriptions of the screens encountered using the TOC-VCSH/CSN/ws are also included in this section.

3.4 Measurement Using the TOC-VCPH/CPN/WP

This section describes procedures for setting analytical parameters and performing calibration curve and sample measurement. Detailed descriptions of the sample tables used with the TOC-VCPH/CPN/WP are also included in this section.

3.1 Analysis Preparations

3.1.1 Sample Boats, Measurement Tools, and Quartz-glass Filter Paper

This section describes the preparation and handling of sample boats, measurement tools and quartz-glass filter paper.

Heat the sample boats in a furnace to oxidize any carbon content. Use an electric furnace of the type normally used in a laboratory.

Note: Tweezers are supplied to handle sample boats. Heat the tip of the tweezers that contacts the sample boat to red hot in the furnace or with gas burner to oxidize any attached carbon.

Preparing the Sample Boat and Quartz-glass Filter Paper

Procedure for TC Analysis

- 1. Heat the sample boat in the furnace at approximately 900°C for 20 minutes. Heat the quartz-glass filter paper in the furnace at approximately 600°C for 20 minutes.
- 2. Store the heat-treated sample boat, quartz glass filter paper and tweezers in a clean container or case.

Procedure for IC Analysis.

- *I*. Soak the sample boat in 2 or 3M hydrochloric acid for about 10 minutes.
- 2. Rinse well with purified water and dry in an oven.

Preparing the Ceramic Fiber

When analyzing a sample that may scatter during combustion, cut off a suitable amount of supplied ceramic fiber and cover the sample with it. Heat the ceramic fiber in the furnace, as described for the sample boat.

Heat the Ceramic Fiber

- I. Cut off a suitable amount of ceramic fiber for a single analysis (approx. 40 to 60mg).
- 2. Shape the ceramic fiber to about 35 mm long x 10mm wide.
- 3. Heat the ceramic fiber, as described for the sample boat.

- 4. Use the heat-treated tweezers to insert the heat-treated ceramic fiber into a clean container or case for storage.
 - Note: The stand used to hold sample boats during analysis should be made of an inorganic material, such as ceramic, glass, or metal, with a surface free from contamination.

Handling

Using a new sample boat for each sample is recommended. However, a sample boat may be reused if the sample analyzed does not contaminate, modify, or degrade the sample boat material. Do not reuse the quartz-glass filter paper.

Reusing a Sample Boat

A used sample boat may appear new but have carbon components or alkaline materials attached to it. Used sample boats must be washed and heated to remove any adhering carbon components before being reused.

Adhering Alkaline Materials

Alkaline materials absorb CO₂ from the atmosphere to form carbonates (such as sodium carbonate and calcium carbonate) which can cause the analysis results to be greater than the actual carbon content in the sample.

.

Sample Boat Cleaning Procedure (Example)

- I. Remove as much of the sample remaining in the used sample boat as possible. Use a brush or the tip of a scraper to scrape off pieces adhering to the bottom of the sample boat.
- 2. Soak the boat in approximately 2M sulfuric acid or hydrochloric acid for about 10 minutes.
- 3. Wash under running tap water for several minutes.
- 4. Rinse the tap water off the boat with de-ionized or distilled water. Dry the boat.
- 5. Bake the dried sample boat in a furnace at approximately 900°C for 20 minutes.
 - *Note:* • Evaluate whether a sample boat can be reused by running a blank test (an analysis with no sample in the boat) to determine the amount of residual carbon.
 - For some samples, it may not be possible to reuse the sample boat even after performing the above treatment procedure. In these cases, use a more appropriate method to remove the residual material from the sample boat or use a new sample boat.

Ceramic Fiber

Ceramic fiber can be reused in cases where a pure product, such as glucose, is measured as the standard.

Although ceramic fiber can be treated according to the above method to remove any contamination, the use of new ceramic fiber is recommended because it is relatively inexpensive.

3.1 Analysis Preparations

3.1.2 Pulverizing the Sample

3.1.2 Pulverizing the Sample

In many cases, the various components in a solid sample are not mixed together uniformly, as they are in liquid samples. For this reason, it is extremely important to homogenize solid samples so as to obtain an aliquot which is as representative as possible of the sample material.

The sampling techniques used should conform to the descriptions in technical literature and the standards established for the corresponding sample type.

Sampling Technique

- 1. Remove any foreign matter that is not the subject of the analysis from the sample material.
- 2. Select portions of the sample from several locations. Thoroughly pulverize and mix these portions in a pulverizer or mortar.
- 3. After pulverizing the sample, pass it through a sieve with a suitable mesh size.
- 4. Repeatedly pulverize any pieces that do not pass through the sieve. Reduce the sample using conical quartering or a riffle sampler.
- 5. Repeat steps 3 and 4 to obtain a final sample that will pass through a 200-mesh sieve.
- **TIP** » *The combustion-oxidation reaction (TC analysis) and the carbonate acidification reaction (IC analysis) are more efficient when the sample particles are smaller in size.*

3.1.3 Drying the Sample

Solid samples may be weighed and measured directly after sampling or they can be dried before they are weighed and measured.

The following three methods are used to dry samples before weighing.

- 1) heating with an electric isothermal dryer
- 1) air-drying over a spread-out sample
- 2) freeze drying

Select the appropriate method for the analytical objectives.

Note: Volatile organic components contained in a sample may be lost during drying.

3.1.4 Pretreatment for Removing IC

The following types of samples cannot be analyzed for IC content by the SSM-5000A and must be pretreated to remove IC content before they can be analyzed for TC content:

- Alkaline samples
 Alkaline substances require a large amount of phosphoric acid to acidify the sample.
- (2) Samples with large particles IC in samples with large particles may not be satisfactorily analyzed due to delayed or incomplete reaction with phosphoric acid.
- (3) Samples with a greater IC content than TOC content

TOC is determined as the difference between measured TC and IC values, and the errors in both the TC and IC analyses are additive. This has a large impact on the TOC result, because TOC cannot be measured precisely.

The pretreatment removal of IC generally consists of reacting the sample with acid. This method provides sufficient time to allow the IC to react with large amounts of acid. In addition, parameters such as heating and agitation of the mixture, can be selected freely to speed up the process, making this method desirable for samples of category (1) and (2) above, as well.

For samples falling in the category of item (3) above, one available method involves pretreatment removal of the IC, followed by measurement based on TC=TOC.

A variety of pretreatment methods are available, including:

- Adding sufficient hydrochloric acid to the sample and heating to remove the IC before drying the sample to remove the excess hydrochloric acid
- · Subjecting the sample to hydrochloric acid gas to remove the IC

For further details, see the literature and test methods that describe such pretreatment methods.

Note: Use a volatile acid, such as hydrochloric acid, for pretreatment. Non-volatile acid may remain in the sample and hinder TC analysis or damage the detector. Include a step to remove the excess hydrochloric acid at the end of the pretreatment.

3.1.5 Loading the Sample Boat

3.1.5.1 Solid Samples

Weighing accuracy is directly reflected in the accuracy of the analysis results. To reduce errors associated with weighing, use an analytical balance or microbalance to carefully weigh the sample directly into a tared heat-treated sample boat.

- *Note:* Reduce the effects of poor distribution, which commonly occur with solid samples.
 - Reduce the weighing error.
 - Reduce the impact of external contamination, by choosing a sample amount that will produce a result close to the upper limit of the instrument (about 30mg C).
- *Note:* Consider the combustion characteristics of each individual sample when determining the amount of sample. Due to a lack of oxygen during combustion, samples that burn extremely rapidly tend to burn less completely with increasing sample size.

Analyzing Crystalline Samples Such as Oxalic Acid or Glucose

Cover the sample boat because these samples may scatter during combustion. If part of the sample scatters out of the sample boat during combustion, artificially low and inaccurate results will be obtained.

Use the supplied ceramic fiber to cover the sample. Ceramic fiber allows good ventilation and is unaffected by heat and oxygen.

Fashion a long, thin cover from ceramic fiber to roughly cover the opening of the sample boat, as shown in Fig. 3.1, and place it over the sample.

Do not cover the sample boat for IC analyses.

IC Analysis of Carbonates

Adding phosphoric acid to a carbonate during IC analysis will cause a violent reaction that may allow part of the sample to scatter outside the sample boat. Pre-moisten this type of sample with a small amount of water.

3.1.5.2 Liquid Samples

The following two sampling methods are available for liquid samples:

- 1) Weighing the sample with a balance
- 2) Measuring the volume of the sample with a microsyringe or pipette
- *Note:* The maximum measurable mass of a liquid sample is 0.5g for TC and 0.3g for IC.
 - Samples that are measured by weighing them on a balance will for the sake of calculation be treated as solid samples by the software.

For TC analysis of a liquid sample, the sample can be impregnated into ceramic fiber placed on the bottom of the sample boat. Ceramic fiber is not used for IC analyses.



3.1.5.3 Swab Samples

Sampling methods for cleaning validation differs depending on the equipment being swab tested. Use the optimal method for the type of equipment being tested.

Sampling Method

- *I*. Heat the quartz-glass filter paper for 20 minutes at approximately 600°C to eliminate any carbon-bearing impurities. Store it in a clean container.
- 2. Use two pieces of the pretreated quartz-glass filter paper, one on top of the other. Use the outside paper to hold the inside paper. (See "Figure 3.2 Cleaning Validation Procedure")
- 3. With the inside filter paper, wipe any residual material from a fixed area of the tested equipment.
- 4. Transfer only the inside filter paper to the pretreated sample boat and analyze.



Figure 3.2 • Cleaning Validation Procedure

TIP » If it is difficult to handle the filter paper by hand, use heat-treated tweezers to hold a single piece of quartz-glass filter paper to be used for swabbing. Use care when swabbing with this method as incomplete swabbing may occur if the filter paper is not held on the equipment surface evenly and with uniform pressure.

3.1.6 Sample Additives

3.1.6 Sample Additives

Sample additives are used with some samples to accelerate combustion or thermal decomposition reactions.

The required amount of sample additive and its effect vary according to the type of sample. Therefore, determine the optimum additive conditions to suit the actual sample.

Use the following commercially available sample additives for the analysis of organic elements, as necessary.

Tungsten Oxide (WO₃)

If a sample contains alkaline metals or alkaline earth metals, carbonates are generated along with carbon or carbon dioxide during combustion. Formation of these carbonates results in lower carbon values because the carbonates do not oxidize during combustion. To prevent this problem, tungsten oxide powder is spread over the entire sample.

Tungsten oxide can also be used as a sample additive for samples containing phosphorous, as they are often inflammable.

Vanadium Pentoxide (V₂O₅)

Vanadium pentoxide is used in the same manner as tungsten oxide for samples containing alkaline metals or alkaline earth metals.

3.1.7 IC Reaction Acid

Undiluted phosphoric acid is normally used as the IC reaction acid. However, for solid samples, reaction with the entire sample will only occur if the entire sample is immersed in phosphoric acid in the sample boat. In some cases, undiluted phosphoric acid does not fully contact the sample due to its high viscosity.

Dilute the phosphoric acid with approximately two parts water to allow the entire sample to come in contact with the acid.

Note: To ensure that all of the CO_2 generated will be carried by the carrier gas, the total volume of dilute phosphoric acid and sample must not exceed 0.5mL.

If a sample consumes an unusually large amount of phosphoric acid (such as with alkaline substances), use pH paper to confirm that the pH of the remaining liquid in the sample boat is pH2 or less.

IC measurement with the SSM-5000A may be not be feasible if the sample consumes too much phosphoric acid.



Phosphoric acid is highly corrosive. Use care to avoid touching or spilling it during handling. See the "Material Safety Data Sheet (MSDS)" section in the TOC-V User Manual for details.

Reference: See Section "3.1.4 Pretreatment for Removing IC".

3.1.8 Standards

The SSM-5000A uses different types of standards depending on the sample type. Some of the typical standards are listed below.

- Government standards that are available from various associations or councils, 1) such as the Marine Sediment Reference Materials available from the National Research Council of Canada.
- 2) Reagents with known compositions and purity, such as glucose and acetoanilide for TC analysis, and sodium carbonate and calcium carbonate for IC analysis.
- 3) Standard solutions obtained by adjusting the reagents in item 2 to desired concentrations.
- 4) Synthetic standards that are prepared by adding the reagents in item 2 or standard solutions in item 3 to actual samples with a known carbon content or with the carbon removed.

The standards used in preparing calibration curves should ideally be of the same type as the sample. If a standard substance described in item 1 is suitable, it should be used. If the carbon content in a sample is known, a pure reagent as in item 2 may be used. A variety of organic reagents are also available commercially for organic element analysis.

Note: Avoid using organic reagents that cause violent combustion. These reagents tend to scatter the sample and increase the likelihood of incomplete combustion.

When samples contain alkaline earths or metals, residual material in the sample boat after combustion will make reusing the sample boat or ceramic fiber difficult without treatment. The potassium hydrogen phthalate used as a TOC-V standard solution contains potassium, which leaves a potassium residue in the bottom of the sample boat. As a result, it would be easier to use glucose instead of potassium hydrogen phthalate with the SSM-5000A.

Precautions for Using a Reagent as a Standard

Since the carbon content of the reagents is quite high (40% for glucose), no more than 30mgC (75 mg glucose) can be used.

Precautions for Analyzing Low-concentration Samples

Prepare a standard solution with a suitable concentration, and introduce it into the sample boat using a microsyringe or pipette. Use a volume of 100μ L or less for water solutions. Larger volumes tend to adversely affect measurement accuracy due to fluctuations in the carrier gas flow rate and a reverse-flow phenomenon when the water vaporizes.

3.1.9 Calibration Curve

3.1.9 Calibration Curve

Due to the difficulty in weighing out the standard and actual sample to exactly equal amounts, calibration curves are created using the carbon amount -- not the carbon concentration -- as the reference.

Atmospheric CO_2 mixes with the sample when the sample port is opened before analysis, which creates a peak. For levels lower than 3.0mg carbon, this peak (SSM-5000A blank peak) cannot be ignored; however, the small blank peak will not affect higher carbon levels.

Measuring Blank Peaks

Blank peaks can be measured in two ways:

- without a sample boat
- with an empty sample boat (in which case the blank peak also contains the blank value of the sample boat)

Blank Peak Compensation

Prepare a multi-point calibration curve containing the blank peak (normally, a two-point calibration curve). This is prepared without using the calibration curve Shift-to-Origin function.

Precautions for Analysis at 3.0mg Carbon or lower

The measurement accuracy decreases with the magnitude of fluctuation in blank peak height. This fluctuation is due to CO_2 in the atmosphere during measurement. To avoid this problem, perform measurements as described below.

TC Measurement

It is possible to measure only the peaks related to the sample because the sample peak appears after the blank peak is complete.

- *1*. Allow carrier gas to flow at a rate of 500mL/min for 1.5 to 2 minutes after closing the sample port cover.
 - *Note:* If the sample contains volatile organic matter, heat from the TC furnace on the sample boat may cause the organic matter to evaporate into the carrier gas.
- 2. Place the sample into the TC furnace for analysis.

IC Analysis

Immediately after the acid is injected into the sample, CO_2 is generated from the reaction with IC in the sample, thereby reducing measurement accuracy.

- 1. Allow carrier gas to flow at a rate of 500mL/min for 1.5 to 2 minutes after closing the sample port cover.
- 2. Inject the acid and immediately insert the sample into the furnace. Start measurement.
Note: In some cases, the TOC-V Ready lamp may go out 60 to 90 seconds after closing the sample port cover. This occurs because the blank peak is evaluated as a base-line disturbance. The Ready lamp illuminates again when the blank peak ends about 2 minutes after closing the sample port cover. After the Ready lamp has illuminated again, insert the sample boat into the furnace and start measurement.

3.1.10 Changing TC Combustion Temperature

Temperature of the SSM-5000A TC furnace is set to 900°C, which is hot enough for satisfactory combustion oxidation of almost any sample in any form. However, measurement of samples containing carbonate (IC component) with a high decomposition temperature, such as calcium carbonate, is sometimes difficult due to slow or incomplete decomposition at 900°C. Such samples exhibit extremely broad peaks or tailing on the screen during measurement. In order to use the SSM-5000A to analyze such samples, the TC furnace temperature setting must be increased to 980°C by following the procedure below.

Note: At 980°C, the life of the catalyst, combustion tube, TC furnace, thermocouple, and 0-ring are shorter than at 900°C.



Verify that the TC and IC furnaces have cooled to room temperature before removing the rear cover to change the temperature setting.

Changing the TC Furnace Temperature Setting

- *I*. Remove the rear cover of the SSM-5000A while the TC furnace and IC furnace are cold.
 - *Note:* Two temperature controllers are located on the right side of the SSM-5000A. The left temperature controller regulates the TC furnace and the controller on the right regulates the IC furnace. The procedure below applies to the TC temperature controller (left).

Reference: See Section "5.6.8.2 Installing the TC Combustion Tube and IC Reaction Tube".

- 2. Move the PROTECTION switch on the TC temperature controller to the down position.
- 3. Use a small stick with a sharp point to press switch ① until the "SP" indicator is illuminated.
 - *Note:* When "SP" is illuminated, the LED displays the set temperature.
 - When "AL" is illuminated, the LED displays the variance range (± °C) for the Ready determination.
 - When neither "SP" or "AL" are illuminated, the LED displays the actual furnace temperature.
- 4. Press switch ③ to set the LED display to 980. The LED display changes quickly if switch ③ is held down. Press switch ② to decrease the temperature setting.

Note: NEVER set the temperature above 980°C.

- 5. Press switch ① until both the "SP" and "AL" indicators are no longer illuminated and the LED displays the actual furnace temperature.
- $\boldsymbol{6}$. Return the PROTECTION switch to the up position.
- 7. Replace the SSM-5000A rear cover.



Figure 3.3 • Temperature Controller

3.1.11 Relationship Between Carbon Content and Peak Height

The approximate relationship between the carbon content of the sample and the peak height when measured with the SSM-5000A is shown below. Refer to these values to check performance after installation or service.

Note: The relationship below was obtained from the measurement of glucose. The relationship may vary depending on the physical and chemical states of the sample.

Table 5.1 • Relationship Between Carbon Content and Peak Height

Analysis Type	Carbon Content	Peak Height	
TC analysis	20mg	Peak height approx. 500mV	

3.2 Start Up

Turn on the power to the TOC-V and SSM-5000A before starting measurement.

3.2.1 Turning on the Power

- *I*. Press the power switch at the bottom-right of the TOC-V front panel. The "Initial Display" is presented.
- 2. Turn on the power switch for the SSM-5000A.
 - *Note:* Before turning on the SSM-5000A, verify that the sample boat push rod knobs are in the CHANGE SAMPLE position.

I ON

0 OFF

I ON

0 OFF

<u>TIP</u> » TOC-V power button positions:

SSM-5000A power button positions:

3.2.2 Carrier Gas Pressure

Use the following procedure to set the TOC-V and SSM-5000A carrier gas pressure.

Setting the TOC-V Carrier Gas Pressure

- I. Set the TOC-V carrier gas supply pressure to 300kPa (44PSI).
- 2. Open the front door of the TOC-V and set the carrier gas pressure to 200kPa using the carrier gas pressure adjustment knob.



Note: The TOC-V carrier gas supply pressure must be at least 300kPa (44PSI) and never more than 600kPa (87PSI).

↔ Setting the SSM-5000A Carrier Gas Pressure

Procedure

- *I*. Set the SSM-5000A carrier gas supply pressure to 300kPa (44PSI).
- 2. Open the SSM-5000A flow controller and use the carrier gas pressure adjustment knob to set the carrier gas pressure to 200kPa.



Figure 3.5 • Setting the SSM-5000A Carrier Gas Pressure

Note: The SSM-5000A carrier gas supply pressure must be set to 300kPa (44PSI).

3.2.3 Setting the Carrier Gas Flow Rate

Use the following procedures to set the TOC-V and SSM-5000A carrier gas flow rates.

Note: Tandem NDIR cells are used for SSM-5000A measurements and carrier gas must flow to both of these cells. Carrier gas must always be supplied to both the TOC-V and the SSM-5000A.

Setting the TOC-V Carrier Gas Flow Rate

Procedure

I. Open the front door of the TOC-V.

2. Turn the carrier gas flow adjustment knob to set the flow on the flow gauge to 150mL/min (TOC-VCSH) or to 130mL/min (TOC-VCSN).



Figure 3.6 • Setting the TOC-V Carrier Gas Flow Rate

Setting the SSM-5000A Carrier Gas Flow Rate

- I. Open the SSM-5000A flow controller.
- 2. Turn the carrier gas flow adjustment knob to set the flow on the flow gauge to 500mL/min.



Figure 3.7 • Setting the SSM-5000A Carrier Gas Flow Rate

Note: Do not change the carrier gas flow rate during analysis. The peak area changes in an inversely proportional relationship to the carrier gas flow rate and changing the flow rate may cause measurement errors.

3.3 Measurements Using the TOC-VCSH/CSN/WS

This section describes the procedures used to perform measurements with the SSM-5000A when it is connected to the TOC-VCSH/CSN/WS.

3.3.1 Analysis Parameters

3.3.1.1 Setting Up the SSM

Enter these parameters before starting SSM analysis.

- *I*. Press the F4 [Conditions] key in the "Initial Display".
 - The "Conditions Unit" screen is displayed.

Basic Unit			Printer	
Furnace Power Tmp	:0N :680	∎ °C	Runtime Report :ON Peak Print :ON Cal Cran Print :ON	
Cat.	∶Regular	Þ		<u>ت</u>
TN			ASI	
Power	:OFF	Þ	yial :	<u></u>
SSM			Reedle Set	<u></u>
SSM Measurement SSM-TC Furnace SSM-IC Furnace Cell Length	t: <mark>OFF</mark> :OFF :OFF :Long		Needle Kince acid add : Stirrer : Flowline wach A: Flowline wach B:	X X
Other Units				
ESU SP Kit	∶Disable ∶Disable	À		
Manual Inj	:Disable	۲		

- 2. Press the arrow keys to move the cursor to the SSM SSM Measurement item.
- *3.* Turn SSM Measurement ON or OFF.

Note: Press the [Select] key to turn the item ON or OFF.

4. Press the F1 [Return] key. The display returns to the "Initial Display".

.

3.3.1.2 Turning on the SSM Electric Furnace

This section describes how to turn on the power to the SSM electric furnace. Turn on the TC furnace for TC measurement or the IC furnace for IC measurement.

TIP » It is possible to turn the TC and IC Furnaces ON even when the SSM measurement item is set to OFF.

The SSM Measurement item must be set to OFF to analyze samples using the TOC-V.

Procedure

I. Press the F4 [Conditions] key in the "Initial Display". The "Conditions - Unit" screen is displayed.

Basic Unit			Printer		
Furnace Power Tmp	:ON :680	°C	Runtime Report Peak Print Cal Gran Print	: ON : ON : ON	À
Cat.	∶Regular	Þ		+011	
TN			Å.N		
Power	:OFF	Þ	Vial.		<u>></u>
SSM			Needle Set	•	<u>}</u>
SSM Measurement SSM-TC Furnace SSM-IC Furnace Cell Length	:ON : ON :OFF :Short	A A A	Needle Kince acid add Stirrer Flowline wach A Flowline wach B		e E
Other Units					
ESU SP Kit	∶Disable ∶Disable	ÀÀ			
Manual Ini	:Disable	×			

- 2. Press the arrow keys to move the cursor to the appropriate item.
 - SSM-TC Furnace item for TC analysis
 - SSM-IC Furnace item for IC analysis
- **3.** Turn the electric furnace ON or OFF.

Note: Press the [Select] key to turn the item ON or OFF.

- 4. Press the F1 [Return] key. The display returns to the "Initial Display".
- *Note:* It takes 40 to 50 minutes for the furnace temperature to stabilize after the furnace power is turned on. Follow the procedures in Section "3.3.6 Ending Measurement" to turn the power off.

3.3.1.3 Setting the SSM Cell Length

This section describes how to select which NDIR detector cell will be used for measurement.

The SSM cell length can be set to "Short" or "Long". "Short" is the default setting for SSM cell length.

Note: Set the SSM cell length item to "Long" for high sensitivity measurement. Use the cell switching valve set (available as a special accessory) to enable high-sensitivity measurement with the 200mm TOC-V cell.

.

Procedure

I. Press the F4 [Conditions] key in the "Initial Display". The "Conditions - Unit" screen is displayed.

Furnace Power Tmp	:ON :680	.C	Runtime Report :ON Peak Print :ON Cal.Grap.Print :ON	A .
Cat.	:Regular	▶		
TN			ASI	
Power	:OFF	Þ	Vial :	<u>8-</u>
SSM			Needle Kince	*
SSM Measurement SSM-TC Furnace SSM-IC Furnace Cell Length	:ON :ON :OFF : <mark>Short</mark>	A A A	Needle Kince acid add : Stirrer : Flowline wach A: Flowline wach B:) N
Other Units				
ESU SP Kit	∶Disable ∶Disable	à		
Manual Inj	:Disable	►		

- 2. Press the arrow keys to move the cursor to the SSM Cell Length item.
- 3. Press the [Select] key to select "Short".
- 4. Press the F1 [Return] key. The display returns to the "Initial Display".

3.3.1.4 Setting the Unit of Concentration

This section describes how to set the unit of concentration for sample measurement. The concentration units are set independently for the standards used to create calibration curves and for sample measurements.

Procedure

1. Press the F4 [Conditions] key in the "Initial Display". The "Conditions - Unit" screen is displayed.

	[-] Basic Unit			Printer	
	Furnace Power Tmp	:0N :680	€	Runtime Report :ON Peak Print :ON Col Core Data : ON The Second Se	
	Cat.	∶Regular	►	Cal.Grap.rrint .UN	
	TN			ASI	
	Power	:OFF	Þ	Vial : ▶ Needle Set : ▶ Neodle Kin∽e : ▶	
	SSM Measuremen SSM-TC Furnace SSM-IC Furnace Cell Length	t:ON : <mark>ON</mark> :OFF :Short		Neetle Kince acid add : M Stirrer : M Flowline wach A: Flowline wach B:	
	Other Units				
	ESU SP Kit	∶Disable ∶Disable	A		
	Manual Inj	:Disable	Þ		
Ret	urn	Un	it	Meas.Cond.	

2. Press the F4 [Meas. Cond.] key. The "Conditions - Meas. Cond." screen is displayed.

l - J Meas.	Output	
<pre># of Syringe Wash :2 # of Syringe Wash(sp):1 Auto Dil change :0FF ■ Auto IC regenerate : IJ Min.Mes.Span[TC/NPOC]:0 sec Min.Mes.Span[IC/POC] :0 sec Name of Method Group</pre>	Unit : Liquid Sample :mg/L Solid Smpl(Cal): Solid Sample :% Name of Convert Val. :CNV Object for Conversion:NONE Equation(y=Ax+B) A:1.0000 B:0.0000	
F2:GROUP-1 F3:GROUP-2 F4:GROUP-3 F5:GROUP-4 F6:CPOUP-5	Misc. Date :12(DEC)-18-2000 14:10 Buzzer :0N Scrn off:0N	Ă

- 3. Press the arrow keys to move the cursor to the appropriate item:
 - Output-Unit-Solid Smpl (Cal) item to set the concentration units for creating calibration curves
 - Output-Unit-Solid Sample item to set the concentration units for sample analysis

4. Press the [Select] key to display the concentration units.

<u>TIP</u> » *Press the [Select] key to interrupt the settings and return to step 3.*

- 5. Press the $\uparrow \downarrow$ keys to select the required units from the following options. Options for Output-Unit-Solid Smpl (Cal):
 - g/L
 - mg/L
 - μg/L
 - ppm
 - ppb
 - %

Options for Output-Unit-Solid Sample:

- g/L
- mg/L
- $\mu g/L$
- ppm
- ppb
- %
- g
- mg
- µg

 $\boldsymbol{6}$. Press the [Enter] key to enter the selected units.

7. Press the F1 [Return] key. The display returns to the "Initial Display".

3.3.2 Calibration Curve Measurement

This section describes how to measure calibration curves.

The example below describes TC analysis with a concentration unit of %, but the same procedure applies to IC analysis.

<u>TIP</u> » Press the [Cal] key to interrupt the settings.

Procedure for Accessing the "SSM Calibration Settings" Screen

Press the [Cal] key in the "Initial Display".

The "SSM Calibration Settings" screen is displayed.

SSM Calibratio	n Settings				Cal.Curves:	1 / 25
SSM-TC♯ 0 1 2 3 4	Conc	Amount	Status ∦ 5 6 7 8 9	Conc	Amount	Status
Calib ♯ : ∎	Amount Unit	∶Weight(mg) d		Cell Length:S	nort
Density :						
[0 - 9] Conc : Amount :	POINT-1 ***** % ***** m(1				
Conc :						
Amount :						
	SSM-TC	SSM-IC				Delete Point

3.3.2.1 Creating Calibration Curves

The method for creating calibration curves can be broken down into the following five steps:

- Step 2 Set the Calibration Curve Measurement Parameters
- Step 3 Load the Standard
- Step 4 Press the [Start] Key
- Step 5 End Measurement

Step 1 Set the Measurement Mode

Procedure

Procedure

I. Press F2 [SSM-TC].

The parameter box for TC analysis is displayed as the default. The currently saved calibration curves are displayed at the top of the screen.

Note: Press the F3 [SSM-IC] key for IC analysis.

Step 2 Set the Calibration Curve Measurement Parameters

The calibration curve measurement parameters include the following items:

- (1) Calibration Curve Number: Enter the number of the calibration curve.
- (2) Amount Unit: Select the form of the standard (solid/liquid).
- (3) Density: Enter the density of liquid standards.
- (4) Concentration: Enter the standard concentration.
- (5) Amount: Enter the weight or volume of the standard.

The procedures for setting each of these items are described below.

.

TIP » Press the F1 [Return to Settings] key to return to the "Setting" screen to repeat entry of the calibration curve parameters. Press the [Cal] key to quit without making the calibration curve measurements. Then press the F6 [Yes] key to quit and return to the "Initial Display" or the F1 [No] key to cancel the quit command.

(1) Calibration Curve Number

I. Enter the calibration curve number. A list of previously saved calibration curves is displayed at the top of the screen.

.

2. Select an unused number from the list.

SSM Cali	brati	on Settin	gs			Cal.Curves:	1 / 25
SSM-	-TC# 0 1 2 3 4	Conc	Amount	Status ∦ 6 7 8 9	Conc	Amount	Status
Calib ♯	:3	Amount U	nit∶Weight(⊮	g) 🖻		Cell Length:	Short
Density	:						
[0 - Conc	^{9]} :	POINT- *****	X				
Amount	:	****	mg				
Conc	:						
Amount	:						
		SSM-TC	SSM-I	C			Delete Point

3. Press the [Enter] key.

(2) Amount Unit

Select whether the standard is a solid or a liquid.

I. Press the \rightarrow key to move the cursor to the Amount Unit item.

	brati	on Setting	S			Cal.Curves:	1 / 25
SSM	-TC# 0 1 2 3 4	Conc	Amount	Status ∦ 6 7 8 9	Conc	Amount	Status
Calib ♯	:3	Amount Ur	it: <mark>Volume(</mark> /			Cell Length:	Short
Density	:	1.00					
[0 - Conc	^{9]} :	POINT-1 ****	%				
Amount	:	****	μL				
Conc	:						
A	:						

- 2. Use the [Select] key to choose the form of the standard.
 - Select Weight (mg) for a solid standard.
 - Select Volume (μL) for a liquid standard.

(3) Density

Density is entered for liquid standards that do not have mg/mL designated as the concentration unit.

- **TIP** » See Section "3.3.1.4 Setting the Unit of Concentration".
 - *I*. Press the \downarrow key to move the cursor to the Density item.

SSM Calibrat	tion Setti	ngs			Cal.Curves:	1 / 25
SSM-TC‡	‡ Conc) 1 3 4	Amount	Status ∦ 5 6 7 8 9	Conc	Amount	Status
Calib ♯∶3	Amount	Unit:Volume(#	L)D		Cell Length:S	hort
Density	: 1.00					
[0 -9999] Conc] : *****	-1 %				
Amount	: *****	μL				
Conc	:					
Amount	:					
L	SSM-T	C SSM-I	c			Delete Point

2. Enter the density and press the [Enter] key.

3.3 Measurements Using the TOC-VCSH/CSN/WS

3.3.2 Calibration Curve Measurement

(4) Concentration

1. Press the \downarrow key to move the cursor to the Concentration (Conc) item.

SSM Calibrati	on Setti	ngs			Cal.Curves:	1 / 25
SSM-TC# 0 1 2 3 4	Conc	Amount	Status ∦ 5 67 89	Conc	Amount	Status
Calib ♯ :3	Amount	Unit:Volume(#	L) 🖻		Cell Length:S	Short
Density :	1.00					
[0 -99999] Conc :	POINT *****	-1 %				
Amount :	****	μL				
Conc :						
Amount :						
l	SSM-T	C SSM-I	C			Delete Point

.

- 2. Enter the concentration of the standard. The previously set concentration value is displayed.
- *3.* Press the [Enter] key.

<u>Reference:</u> See Section "3.3.1.4 Setting the Unit of Concentration".

(5) Amount

1. Press the \downarrow key to move the cursor to the Amount item.

SSM Calibrati	on Settin	gs			Cal.Curves:	1 / 25
SSM-TC# 0 1 2 3 4	Conc	Amount	Status ♯ 5 6 7 8 9	Conc	Amount	Status
Calib ♯ ∶3	Amount U	nit:Volume(µ]	L) 🖻		Cell Length:S	hort
Density :	1.00					
[0.001-9999] Conc : Amount :	POINT- 1.000 *****	μ <u>μ</u>				
Conc :						
Amount :						
	SSM-TC	SSM-I	;]			Delete Point

2. Enter the amount of standard and press the [Enter] key. Proceed to Step 3 "Load the Standard" if creating a single-point calibration curve or follow the procedure below to create a multi-point calibration curve.

Creating a Multi-point Calibration Curve

- *I*. Move the cursor to the Concentration (Conc) item.
- 2. Press the \rightarrow key. The parameter box is displayed for the second point.

SSM Calibratio	on Settings				Cal.Curves:	1 / 25
SSM-TC# 0 1 2 3 4	Conc ,	Amount	Status # 5 6 7 8 9	Conc	Amount	Status
Calib ♯:3	Amount Unit	∶Volume(µL)	Þ		Cell Length:S	nort
Density :	1.00					
[0 -99999] Conc : Amount :	POINT-1 1.000 % 100.0 //L	POINT-2 ***** *****	% //L			
			<u>.</u>			
Conc :						
Amount :						
3	SSM-TC	SSM-IC				Delete Point

- 3. Set the parameters for the second point in the same way as parameters for the first point were set.
- 4. Repeat steps 2 and 3 to set the parameters for the third and subsequent points in the calibration curve.

To Delete a Point (point n)

- I. Move the cursor to the parameter box for point n.
- 2. Press the F6 [Delete Point] key. The parameter box for point n is deleted.
 - *Note:* If the F6 [Delete Point] key is pressed while the parameter box for the first point (point 1) is selected by the cursor, this parameter box is not deleted but the display reverts to the default display.
- **TIP** » Press the F1 [Return to Settings] key to return to the "Setting" screen to repeat entry of the calibration curve parameters. Press the [Cal] key to quit without making the calibration curve measurements. Then press the F6 [Yes] key to quit and return to the "Initial Display" or the F1 [No] key to cancel the quit command.

Step 3 Load the Standard

Procedure

- *I*. Press the [Next] key.
 - The "SSM Calibration" screen is displayed.

SSM Calibration			
Calib ♯ : 3	OINT-1		_
Mean Area SD CV%		Input the volume of next sample. [0.001-9999]	
1000 mV			πν Area Vol(μL) Remark
SSM TC		- 4: 5: 6: 7: 8:	
-			
0 <u>.</u> 0 10 Pro	20 tect:OFF Le	Cell Length 30(min) east Squares Shift:none	12(DEC)-18-2000 14°33 a:Short Densit:1.00 e
Return	Z	Zoom In Zoom Out	

- If the standard is a solid, a dialog box prompts the operator to enter the weight of the standard in mg.
- If the standard is a liquid, a dialog box prompts the operator to enter the volume of the standard in μL .
- 2. Weigh the standard into the sample boat. Enter the amount of the standard in the sample boat and press the [Enter] key.
- 3. Press the [Enter] key again. A dialog box instructs the operator to load the *** mg/L Standard Set Sample and press the START button.

*** Represents the standard concentration entered in the "SSM Calibration Settings" screen.

- 4. Open the sample port cover.
- 5. Place the sample boat containing the standard on the sample boat holder.
- 6. Close the sample port cover completely. When the instrument is in standby, the Ready lamp illuminates and the [Start] key flashes.
 - **Note:** If the carbon amount in the standard (concentration x amount) is less than 3.0mg, wait 1.5 to 2 minutes after closing the sample port cover before pressing the [Start] button to avoid peaks due to ambient CO_2 .
- **TIP** » Press the F1 [Return to Settings] key to return to the "Setting" screen to repeat entry of the calibration curve parameters. Press the [Cal] key to quit without making the calibration curve measurements. Then press the F6 [Yes] key to quit and return to the "Initial Display" or the F1 [No] key to cancel the quit command.

Step 4 Press the [Start] Key

Reference: The parameters used to create the calibration curve can be changed on the "SSM Calibration" screen during the calibration curve measurement or on the "SSM Calibration Setting" screen after calibration curve measurement is complete. See Section "3.3.2.2 Changing Calibration Curve Parameters".

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Procedure

1. Verify that the Ready lamp is illuminated and the [Start] key is flashing.

2. Press the [Start] key.

A dialog box prompts the operator to move the standard to the MEASURING position.

SSW Calibration Calib # : 3 Conc. 1.000% Mean Area SD CV%	Push boat to Measuring Position.	
50 mV SSM TC 0	Inj# Area Cnv Area Vol(#L) Remar 2: 3: 4: 5: 6: 7: 8: 9: 10: 12(DEC)-18-2000 14. Cell Length:Short Densit:1.	k■ 34 00
Protect: Measurement Ready St Conditions	UFF Least Squares Shift:none atus Zoom In Zoom Out Browse Cal. Curves	

- 3. Move the sample boat into the furnace.
 - TC analysis: Gently move the sample boat push rod knob to the MEASURING position (until it stops).
 - IC analysis: Inject the acid into the sample boat using the acid dispenser, then gently move the sample boat push rod knob to the MEASURING position (until it stops).

The [Start] key illuminates and calibration curve measurement starts.

<u>TIP</u> *» Graph Scale*

The graph scale (vertical axis) is automatically displayed. The range can be changed between 10 and 1000mV with the F3 [Zoom In] and F4 [Zoom Out] keys. The scale changes by 10mV each time the F3 and F4 key is pressed in the 10-100mV range and by 100mV each time the key is pressed in the 100-1000mV range.

3.3.2 Calibration Curve Measurement

Procedure After Analysis

After analysis is complete the following instructions are displayed.

- TC analysis: Return the standard to the COOLING position.
- IC analysis: Return the standard to the SAMPLE CHANGE position. Proceed to procedure step 2 below.
- 1. Move the sample boat push rod knob to the COOLING position. The sample boat cools down. About 30 seconds later, the display instructs the user to return the standard to the SAMPLE CHANGE position.
 - *Note:* Damage may occur if the TC sample boat is moved directly from the MEASURING position to the SAMPLE CHANGE position.
- 2. Return the sample boat push rod knob to the SAMPLE CHANGE position.
- **3.** Proceed to the next step.
 - Press the F1 [Add Injection] key for additional measurements of the same standard. Steps 3 "Load the Standard" and 4 "Press the [Start] Key" are repeated.
 - Press the F2 [Remeasure Point] key to delete the data and repeat the measurement. Then press the F6 [Yes] key to repeat steps 3 "Load the Standard" and 4 "Press the [Start] Key".
 - Operation proceeds to step 5 "End Measurement" when measurement is complete.
 - Press the F6 [Go to Next Cal. Point] key to proceed to the measurement of the next concentration (for multi-point calibration curve). Steps 3 "Load the Standard" and 4 "Press the [Start] Key" are repeated.

Reference: See Section "3.3.5 Deleting Data" before deleting any measured data.

Step 5 End Measurement

Procedure

- *I*. Press the F6 [Exit Cal. Measurement] key.
- 2. Press the appropriate key as desired:
 - Press the F6 [Yes] key to protect the results. The measurement results are automatically printed out if the Runtime Report item was set ON in the "Conditions Unit" screen. The display returns to the "Initial Display".
 - Press the F4 [No] key if the results are not to be protected. The measurement results are automatically printed out if the Runtime Report item was set to ON in the "Conditions Unit" screen. The display returns to the "Initial Display".
 - Press the F1 [Cancel] key to return to the "Measuring" screen without ending measurement. The display returns to the "SSM Calibration" screen.
 - *Note:* Occasionally, IC reaction acid overflows from the sample boat and accumulates on the IC sample boat holder. During sample changes, inspect and clean the sample boat holder as necessary.
- **Reference:** See Section "3.3.7.1 Calibration Curve List" when changing the general parameters for the calibration curves after measurement is complete.

3.3.2.2 Changing Calibration Curve Parameters

The following three calibration curve parameters can be changed during or after calibration curve measurement:

(1) Protection

Select whether the stored calibration curve data is protected. Protected calibration curve data cannot be deleted.

(2) Curve Type

Select whether calibration curves with 3 or more points are created using regression lines by the method of least squares (Least Squares) or using the polygonal method linking consecutive points with straight lines (Pt to Pt).

(3) Curve Shift

Select whether the calibration curve is parallel shifted to pass through the origin.

Note: The default parameters for generating calibration curves are as follows.

- Protection: OFF
- Curve Type: Least Squares
- Curve Shift: none

Procedure

- *I*. Press the F5 [Browse Cal. Curves] key.
- 2. Change the calibration curve parameters.
 - Protection: Press the F2 key to choose ON or OFF.
 - Curve Type: Press the F3 key to choose Least Squares or Pt to Pt.
 - Curve Shift: Press the F4 key to choose Origin or none.

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3.3 Measurements Using the TOC-VCSH/CSN/WS

3.3.3 Sample Measurement

3.3.3 Sample Measurement

Sample measurement is possible either with just one measurement mode (single-mode measurement) or with more than one measurement mode (multiple-mode measurement).

The example below describes TC analysis with concentration units of %, but the same procedure applies for IC analysis.

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Procedure for Accessing the "SSM Measurement Settings" Screen

Press the [Measure Sample] key in the "Initial Display".

The "SSM Measurement Settings" screen is displayed.

SSM Meas	sureme	nt Setti	ngs		(Cal.Curves:	2 / 25
	#0 12 34	Conc	Amount	Status # 5 6 7 8 9	Conc	Amount	Status
Sample	Name:			Amount Unit:W	eight 🖻	Cell L	ength∶Short
[- Calib]:						
Density	ı :						
		CCM-T	d GdW-	10			
			. ויומט 🌆 ט				

TIP » *Press the [Measure Sample] key to interrupt the settings and return to the "Initial Display" screen.*

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3.3.3.1 Measurement Procedure

The method for measuring samples can be divided into the following five steps:

- Step 1 Set the Measurement Mode
- Step 2 Set the Measurement Parameters
- Step 3 Load the Sample
- Step 4 Press the [Start] Key
- Step 5 End Measurement

☆ Step 1 Set the Measurement Mode

Procedure

- *I*. Press the F2 [SSM-TC] key.
 - The parameters box for TC analysis is displayed.

SSM Measu	remen	t Setting	S		C	al.Curves:	8 / 25
	# 0 1 2 3 4	Conc.	Amount	Status # 5 7 8 9	Conc.	Amount	Status
Sample N	ame:			Amount Unit:W	leight∎	Cell Le	ngth:Short
[- Calib.] :	SSM-TC # ****	*				
Density	•						
1			× SSM-	-IC			

Note: Press the F3 [SSM-IC] key for IC analysis.

<u>TIP</u> » *Press the function key corresponding to the measurement mode to hide the parameter box for that measurement mode.*

Step 2 Set the Measurement Parameters

The measurement parameters include the following items:

- (1) Sample Name: Enter the name of the sample.
- (2) Sample Amount Unit: Enter the form of the sample (solid/liquid).
- (3) Calibration Curve: Enter the calibration curve to be used.

The procedure for setting each of these items is described below.

(1) Sample Name

I. Enter the name of the sample using up to 16 characters.



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2. Press the [Enter] key to confirm the sample name.

(2) Amount Unit

The form of the sample (solid or liquid) is entered in this field.

I. Press the \rightarrow key to move the cursor to the Amount Unit item.

SSM Measu	remen	t Setting	IS			Cal.Curves:	8 / 25
	#0 1 2 3 4	Conc.	Amount	Status # 5 6 7 8 9	Conc.	Amount	Status
Sample Na	ame:S	AMPLE-1		Amount Unit:	′olume⊡	L Cell	Length:Short
[- Calib.]:	SSM-TC # ****	*				
Density	•	1.00					
			× SSM-	-IC			

- 2. Use the [Select] key to choose the form of the sample.
 - Select Weight for a solid sample.
 - Select Volume for a liquid sample.

(3) Calibration Curve

I. Press the \downarrow key to move the cursor to the Calibration Curve (Calib.) item. A list of TC analysis calibration curves is displayed at the top of the screen.

som measureme	nt settings				cal.curves.	IV 7 ZJ
SSM-TC# 0 1 2 3 4	Conc. 1.000% 2.000%	Amount 100.0mg 100.0mg	Status # 5 P 6 7 8 9	Conc.	Amount	Status
Sample Name: [0 - 9] Calib. : Density :	SAMPLE-1 SSM-TC ****** 1.00	X	Amount Unit:V	olume 🖻	Cell)	.ength:Short
Calibration Curv	e	× SSM-	IC			

- 2. Select the calibration curve to be used by one of the following two methods:
 - Select it from the "SSM Measurement Settings" screen.
 - Select it from the calibration curve display.

Selecting from the "SSM Measurement Settings" Screen

- I. Enter the calibration curve number from the list at the top of the screen.
- 2. Press the [Enter] key.

The concentration and injection volume that correspond to the selected calibration curve are displayed.

Selecting from the Calibration Curve Detailed Display

I. Press the F1 [Calibration Curve] key to display the calibration curve details.



- 2. Press the $\uparrow \downarrow$ keys to select the calibration curve from the list at the top of the screen.
- **3.** Press the F6 [Select] key.

The display reverts to the "SSM Measurement Settings" screen. The concentration and injection amount that correspond to the selected calibration curve are displayed.

Note: • For single mode measurement: Proceed to step 3 "Load the Sample".

• For multiple mode measurement: Repeat the procedure from step 1 "Set the Measurement Mode".

↔ Step 3 Load the Sample

Procedure

- *I*. Press the [Next] key.
 - The "SSM Measurement" screen is displayed.



- If the sample is a solid, a dialog box prompts the operator to enter the weight of the sample in mg.
- If the sample is a liquid, a dialog box prompts the operator to enter the volume of the sample in μL .
- 2. Measure the sample into the sample boat. Enter the amount of the sample in the sample boat and press the [Enter] key.
- 3. Press the [Enter] key again.
 - A dialog box instructs the operator to load the sample and press the [Start] button.
- 4. Open the sample port cover.
- 5. Place the sample boat (with sample) on the sample boat holder.
- 6. Close the sample port cover completely. When the instrument is in standby, the Ready lamp is illuminated and the [Start] key flashes.
- **TIP** » Press the F1 [Return] key to return to the "Setting" screen to repeat the sample measurement settings. Press the [Measure Sample] key to quit without making the sample measurement and return to the "Initial Display".
 - **Note:** If the carbon amount in the sample (concentration x amount) is 3.0mg or less, wait 1.5 to 2 minutes after closing the sample port cover before pressing the [Start] button to avoid peaks due to ambient CO_2 .

↔ Step 4 Press the [Start] Key

Procedure

- *I*. Verify that the Ready lamp is illuminated and the [Start] key is flashing.
- 2. Press the [Start] key.

A dialog box prompts the operator to move the sample to the MEASURING position.

SSM	Measurement						
	Sample NameSAM	PLE-1 SM-TC					
	Conc. SD CV% TOC Conc. CNV	Pus	sh boat to Measu	ring Po	osition.		
601	WV			Inj∦ 1:	Area (onc. Vol(µ	L) Remark
SSI TC	M			2: 34: 5:			
				7: 8: 9: 10:			
	0 <u>.</u> 0 10	20		Cell	1 Length:Sho	2(DEC)-19- rt	2000 16°18 Densit:1.00
M	easurement Rea Conditions	dy Status Zo	oom In	Zoom	Out		

- 3. Move the sample boat into the furnace.
 - TC analysis: Gently move the sample boat push rod knob to the MEASURING position (until it stops).
 - IC analysis: Inject the acid into the sample boat using the acid dispenser then gently move the sample boat push rod knob to the MEASURING position (until it stops).

The [Start] key illuminates and sample measurement starts.

<u>TIP</u> *w Graph Scale*

The graph scale (vertical axis) is automatically displayed. The range can be changed between 10 and 1000mV with the F3 [Zoom In] and F4 [Zoom Out] keys. The scale changes by 10mV each time the F3 and F4 key is pressed in the 10-100mV range and by 100mV each time the key is pressed in the 100-1000mV range.

Procedure After Analysis

After analysis is complete, the following instructions are displayed.

- TC analysis: Return the sample to the COOLING position.
- IC analysis: Return the sample to the SAMPLE CHANGE position. Proceed to procedure step 2 below.
- 1. Move the sample boat push rod knob to the COOLING position. The sample boat cools. About 30 seconds later, the display instructs the user to return the sample to the SAMPLE CHANGE position.
 - *Note:* Damage may occur if the TC sample boat is moved directly from the MEASURING position to the SAMPLE CHANGE position.
- 2. Return the sample boat push rod knob to the SAMPLE CHANGE position.
- 3. Proceed to the next step.
 - Press the F1 [Add Injection] key for additional measurements of the same sample. Steps 3 "Load the Sample" and 4 "Press the [Start] Key" are repeated. Operation proceeds to step 5 "End Measurement" when measurement is complete.
 - Press the F6 [Next Measurement] key to proceed to the measurement of the next mode (for multiple mode measurement). Steps 3 "Load the Sample" and 4 "Press the [Start] Key" are repeated.

☆Step 5 End Measurement

Procedure

- 1. Press the F6 [Exit] key to return to the "Initial Display" and save the measurement results in the data report.
 - *Note:* The measurement results are automatically printed out if the Runtime Report item was set to ON in the "Conditions Unit" screen.

3.3 Measurements Using the TOC-VCSH/CSN/WS

3.3.4 Stopping Measurement

3.3.4 Stopping Measurement

Two methods are available to stop in-progress measurement.

(1) HALT

Measurement stops immediately and measured data is lost.

(2) PEAK STOPPeak measurement stops immediately and measured data to that point is displayed.

Procedure

- *1*. Press the [Stop] key during measurement. The following function keys are enabled:
 - F1 key: Cancel (Do <u>not</u> stop measurement.)
 - F3 key: HALT
 - F6 key: Peak Stop
- 2. Press the appropriate function key.

Note: • Measurement stops immediately when the F3 [HALT] key or F6 [Peak Stop] key is pressed.

When TC analysis is stopped, instructions are displayed to return the sample to the COOLING position.

When IC analysis is stopped, instructions are displayed to return the sample to the SAMPLE CHANGE position. Proceed to procedure step 4 below.

- **3.** Move the sample boat push rod knob to the COOLING position. The sample boat cools. After about 30 seconds, the screen displays instructions to return the sample to the SAMPLE CHANGE position.
 - *Note:* Damage may occur if the TC sample boat is moved directly from the MEASURING position to the SAMPLE CHANGE position.
- 4. Return the sample boat push rod knob to the SAMPLE CHANGE position. A dialog box is displayed verifying whether to end measurement.
- 5. Press the F6 [Exit Measurement] key. The display returns to the "Initial Display".
 - *Note:* Press the F1 [Add Injection] key to repeat the measurement.

3.3.5 Deleting Data

To improve reliability of measurement results when creating calibration curves or analyzing samples, measurement is normally repeated several times on the same sample. If large discrepancies in measured data values result, abnormal values can be deleted.

Note: Data cannot be deleted after measurements are complete and the "Measuring" screen has been closed.

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Deleting Data

- 1. Verify that measurements are complete and the measurement results are displayed in the list at the right of the screen.
- 2. Press the $\uparrow \downarrow$ keys to select the data to be deleted.
- 3. Press the [CE] key. The data is deleted. "Exc" is displayed before "Inj#". The mean area, SD, and CV% values show at the top of the screen are updated.

Restoring Deleted Data

- *I*. Press the $\uparrow \downarrow$ keys to select the deleted data to be restored.
- 2. Press the [CE] key.

"Exc" disappears and the mean area, SD, and CV% values shown at the top of the screen are updated.

3.3.6 Ending Measurement

The end of a measurement session can be initiated by either of the following two methods:

(1) Power off

Turn off the instrument power supply.

(2) Sleep

Set the date and time of the next measurement and leave the instrument in an inactive condition until then.

- Note: Always turn off the SSM-TC furnace power supply at least 30 minutes before turning off the SSM-5000A power supply. If the SSM-5000A is turned off immediately after turning off the SSM-TC furnace power supply, the interior of the furnace remains hot but the cooling fan for the TC combustion tube connector stops, allowing the O-ring temperature to rise in the TC combustion tube connector and reducing the O-ring life. This restriction does not apply to emergency shutdowns.
 - Prior to leaving the SSM-5000A unused for a long period, use water to eliminate or flush out the phosphoric acid from the tubing between the acid dispenser and the IC sample port. Phosphoric acid left in the tubing may solidify and form a blockage.
 - Use a cotton swab to remove any acid or other corrosive substance remaining in the TC or IC sample ports (sample boat holder and sample boat push rod included) or on internal parts of the combustion tube swage lock connector. Wash off any particularly corrosive substances with water.

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Turning Power Off

Use the procedure below to end measurement and turn off the instrument power.

Procedure

1. Press the F1 [Standby Option] key in the "Initial Display". The "Standby Option - Sleep" screen is displayed.

1	An. 1	_							
standby	Uption								
		Standhu On	tion						
		Didnubg op	01011						
		Nov+	nohoo+	• 10/nc	d)_00_000	0 16+00			
		next	Tennor	• 12 (DE	07 20 200	0 10+20			
			11	1. Canali	1.1.4.4.4				
			UN	it Condi	tions				
					ARR				
			Furnace		UFF 🖻				
			Carrier	Gas	OFF 🗉				
			TN Unit		OFF 🖭				
				r					
Cancel				S	leep	[vecute	
					1000	Power	OFF		
						1000 CI			

2. Press the F4 [Power OFF] key.

A message indicates that the instrument is entering the Automatic Shutdown process.

Standby Uption						
Standby Optio	n					
	Automatic	Shutdow	n			
	AUTOMOTIC	bilataow				
Cancel		<u></u>		Power	r OFF 📗	Execute
		Sle	ep			
					-	

- **<u>TIP</u>** » *Press the F1 [Cancel] key to cancel turning off the power and return to the "Initial Display".*
 - 3. Press the F6 [Execute] key.

A dialog box prompts whether to turn off the power.



- 4. Press the F6 [Yes] key to end. The furnace power is automatically shut off and the temperature drops.
- 5. After 30 minutes the TOC-V power automatically turns off. Turn off the SSM-5000A power after the TOC-V turns off.

Sleep

Set the time and date for the next measurement session. The TOC-V stays in an inactive state until then.

About 1 hour before the preset date and time, the TOC-V prepares for measurement. The power is turned on, the carrier gas flow is started and the electric furnace temperature rises.

The furnace power and carrier gas can be turned off during the inactive period.

Power to the SSM-5000A electric furnaces are controlled by the TOC-V. The carrier gas for the SSM-5000A is turned ON and OFF manually. If the SSM-5000A is going to be turned ON automatically by the Sleep function, the carrier gas flow rate to the SSM-5000A must remain ON during the inactive period.

Procedure

- *I*. Press the F1 key in the "Initial Display".
 - The "Standby Option Sleep" screen is displayed.

Standhu Ontion		1				
Didhaba Option						
Star	ndby Option					
	Next reboot	: 12(DEC)	-20-2000	16:20		
	Un	it Condit	ions			
	Furnace Carrier TN Unit	Gas	OFF OFF OFF			
Cancel		Sle	eep	Power OF	F Execu	te

- 2. Enter the desired date and time of the next instrument startup.
- **TIP** » The date and time can be set between the present and December 31, 2099. Press the \rightarrow key to move the cursor to the right and the \leftarrow key to move the cursor to the left. The cursor moves in the sequence: month, day, year, hour, minute. Press the [Enter] key to confirm each numerical value.
 - 3. Press the \downarrow key to move the cursor to the unit to be set, then press the [Select] key to toggle the setting ON or OFF.
 - *Note:* The term "Furnace" refers to the SSM-TC furnace, SSM-IC furnace, and TOC-V furnace.
 - The carrier gas ON/OFF setting affects only the TOC-V carrier gas setting. Turn the SSM-5000A carrier gas ON and OFF manually.

TIP » Press the F1 [Return] key to cancel turning off the power.

4. Press the F6 [Execute] key.

The units set to OFF automatically turn off. These units will turn on approximately 1 hour before the preset time, so that the instrument is ready for measurement.

3.3.7 Referencing Data

Previously saved data (calibration curves and data reports) can be referenced and the parameters can be changed.

3.3.7.1 Calibration Curve List

It is possible to reference calibration curve details, change curve parameters (protection, curve type, curve shift), and delete previously saved calibration curves.

Calibration Curve Details can be referenced and the parameters can be changed during calibration curve measurement. (See Section "3.3.2 Calibration Curve Measurement".) Saved Calibration Curve Details can be referenced when setting up sample measurements. (See Section "3.3.3 Sample Measurement".)

The example in this section describes procedures for referencing TC analysis calibration curves but the same procedure applies to IC analysis calibration curves.

Referencing the Calibration Curve Details

Procedure

1. Press the F6 [Data Report] key in the "Initial Display". The "Data Report - Measurement Log" screen is displayed.

Data Report - Mea:	sure	ement Log						1/1
<u>Sample Name</u>	V#	Date & Time	Type	Conc.	SD	CV%	TOC	
SAMPLE-1	1	DEC/19/00 18:23	SSM-TC	0.854%	0.00	0.00		
Return			Clea	ar All		C	al. Cu	rves
			0100				ort ou	T lot

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2. Press the F6 [Cal. Curves List] key. The "Data Report - Calibration Curves" screen is displayed.



3. Press the F2 [TC] key. The saved TC calibration curves are displayed by default.

Note: Press the F3 [IC] key to list the IC analysis calibration curves.

- 4. Press the $\uparrow \downarrow$ keys to select the calibration curve to be referenced. The calibration curve graph and details are displayed.
- 5. Press the F1 [Return] key to return to the "Initial Display".

Changing Calibration Curve Parameters

The following three calibration curve parameters can be changed:

- Protection
- Curve Type
- Curve Shift

See Section "3.3.2.1 Creating Calibration Curves" for details.

Procedure

1. Press the F6 [Data Report] key in the "Initial Display". The "Data Report - Measurement Log" screen is displayed.



- 2. Press the F6 [Cal. Curves List] key.
 - The "Data Report Calibration Curves" screen is displayed.





3. Press the F2 [TC] key.

The saved TC analysis calibration curves are displayed by default.

Note: Press the F3 [IC] key to list the IC analysis calibration curves.

- 4. Press the $\uparrow \downarrow$ keys to select the calibration curve to be changed. The calibration curve graph and details are displayed.
- 5. Press the F6 [Edit] key.
 - The "Data Report Calibration Curves" screen is displayed.



- $\boldsymbol{6}$. Change the calibration curve parameters.
 - Protection: Press the F2 key to choose ON or OFF.
 - Curve Type: Press the F3 key to choose Least Squares or Pt to Pt.
 - Curve Shift: Press the F4 key to choose Origin or none.
- 7. Press the F6 [OK] key. Status reverts back to the status in step 3.
- **<u>TIP</u>** » *Press the F1 [Cancel] key to cancel the change operation.*
 - δ . Press the F1 [Return] key to revert to the "Initial Display".
Deleting Calibration Curves

Procedure

1. Press the F6 [Data Report] key in the "Initial Display". The "Data Report - Measurement Log" screen is displayed.



2. Press the F6 [Calibration Curve List] key. The "Data Report - Calibration Curve" screen is displayed.



3. Press the F2 [TC] key.

The TC analysis calibration curves saved by default are displayed.

Note: Press the F3 [IC] key to list the IC analysis calibration curves.

4. Press the $\uparrow \downarrow$ keys to select the calibration curve to be deleted. The calibration curve graph and details are displayed.

5. Press the [CE] key.



- Press the F6 key to delete the selected calibration curve.
- Press the F1 key to cancel the delete operation.

6. Press the F1 [Return] key to revert to the "Initial Display".

If a Calibration Curve Cannot be Deleted

A calibration curve cannot be deleted if the letter "S" or "P" is displayed in its status column. An error message is displayed after the [CE] key is pressed.

Use the methods below to change the calibration curve status.

- "S" displayed: The letter "S" displayed in status column indicates that the calibration curve is being used in the "Measurement - Settings" screen. Press the [Measure Sample] key to hide the parameter box or deselect the calibration curve with the Calibration Curve item. (See Section "3.3.2.1 Creating Calibration Curves".)
- "P" displayed: The letter "P" displayed in status column indicates that the calibration curve is protected. Turn the Protect OFF. (See Section "3.3.2.2 Changing Calibration Curve Parameters".)

3.3 Measurements Using the TOC-VcsH/csN/ws 3.3.7 Referencing Data

3.3.7.2 Data Reports

This section describes how to reference or delete measured sample data.

Referencing Data

1. Press the F6 [Data Report] key in the "Initial Display". The "Data Report - Measurement Log" screen is displayed.



Deleting Data

I. Press the F4 [Clear All] key in the "Measurement Log" screen to delete the data. A dialog box prompts whether to delete the measured data.



- 2. Press the F6 [Yes] key. All stored data is deleted and the data display is cleared.
- **<u>TIP</u>** » Press the F1 [No] key to cancel the delete operation and return to the "Measurement Log" screen.
 - 3. Press the F1 [Return] key to revert to the "Initial Display".

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3.3.8 Screen Displays

3.3.8.1 Background Monitor

Press the F3 key in the "Initial Display" to display the "Background Monitor" screen.



Key to the Display

- (1) Screen title: Background Monitor
- (2) Baseline display
- (3) TOC-V furnace temperature and OK/NG status
- (4) TOC-V dehumidifier (electronic cooler) temperature and OK/NG status
- (5) Baseline position, fluctuation, and noise OK/NG status
- (6) SSM-5000A TC furnace temperature and OK/NG status
- (7) SSM-5000A IC furnace temperature and OK/NG status

Function Keys

Key	Function
F1 [Return]	Returns to the "Initial Display".
F3 [Zoom In]	Enlarges the baseline full scale to 20mV.

3.3.8.2 Conditions

This section describes only the items related to the SSM-5000A on the "Conditions" screen.

The "Conditions" screen comprises two screens: "Unit" and "Measurement Conditions (Meas. Cond.)".

☆"Conditions - Unit"

Press the F3 "Unit" key to display the following screen.

	[-] Basic Unit			Printer		
	Furnace Power Temp.	:0N :720	€	Runtime Report : Peak Print	: ON : ON	
	Catalyst Type	:TC/TN	Þ	Val.0rap.frint -	• ON	
	TN			ASI		
1.	Power	:ON	Þ	Vial Needle Set	: 40mL	
	SSM			Needle Rinse	:0N	
	SSM Measuremen SSM-TC Furnace SSM-IC Furnace Cell Length	t: <mark>ON</mark> :OFF :OFF :Short		After acid add Stirrer Flowline wash A Flowline wash B	: ON : OFF : 1 : 1	
	Other Units			1		
	ESU SP Kit	:Disable :Disable	À			
	Manual Inj	:Disable	Þ			
Re	turn	Un	it	Meas.Cond.]	J

Key to the Display

(1) SSM

-

Item	Description
SSM Measurement	Turns SSM-5000A measurement ON or OFF.
SSM-TC Furnace	Turns the SSM-5000A TC furnace ON or OFF.
SSM-IC Furnace	Turns the SSM-5000A IC furnace ON or OFF.
Cell Length	Set to "Long" or "Short" to designate whether the long or short tandem NDIR detector cell is used for SSM-5000A measurements. The "Short" is the default setting for the SSM-5000A.

* "Conditions - Meas. Cond."

Meas.	Output	
♯ of Syringe Wash :2 ♯ of Syringe Wash(sp):1	Unit…Liquid Sample :mg/L Solid Smpl(Cal):K Solid Sample :%	
Auto Dil change :ON ₪ Auto IC regenerate :ON ₪	Name of Convert Val. :CNV Object for Conversion:TOC(TC-IC) Equation(y=Ax+B) A:1.0000 B:0.0000	
Min.Mes.Span[TC/NPOC]:O sec Min.Mes.Span[IC/POC]:O sec		
Name of Method Group		
F2:GROUP-1 F3:GROUP-2	Misc.	
F4: GROUP-3 F5: GROUP-4 F6: GROUP-5	Date :12(DEC)-19-2000 19:06 Buzzer :0FF Scrn off:0FF	Þ

Press the F4 [Meas. Cond.] key to display the following screen.

Key to the Display

(1) Output

Item

Description

Unit-Liquid Sample	Select the concentration unit for liquid samples.
Unit-Solid Smpl (Cal)	Select the concentration unit for the calibration curves.
Unit-Solid Sample	Select the concentration unit for solid samples.

3.3.8.3 "Data Report - Measurement Log"

This section describes the "Data Report - Measurement Log" screen, which shows the results history of previously conducted measurements. Each measurement type item is displayed on a single line. Up to 200 measurement results can be saved. Up to 25 measurement results can be displayed on each page.



Key to the Display

Item	l	Description
(1)	Sample name	Name of the measured sample.
(2)	V#	0 is displayed for SSM measurements.
(3)	Date & Time	Date and time of the measurement.
(4)	Туре	Measurement type.
(5)	Conc.	Concentration analysis value.
(6)	SD	SD of measurement results.
(7)	CV%	CV% of measurement results.
(8)	TOC	TOC concentration calculated as TOC = TC-IC. Displayed in the IC position when TC and IC measurements are made.
(9)	Page number	Current page number is displayed before the slash (/) and the total number of pages is displayed after the slash. To switch pages, move the cursor to the bottom of the page and press the \downarrow key or move the cursor to the top of the page and press the \uparrow key.

Function Keys

Key	Function
F1 [Return]	Returns to the "Initial Display".
F4 [Clear All]	Deletes the measurement history.
F6 [Cal. Curves List]	Displays the "Data Report - Calibration Curves" screen.

3.3.8.4 "Data Report - Calibration Curves"



This section describes the "Data Report - Calibration Curves" screen.

✤Display in Editing Mode

This screen is used to check and edit calibration curve data.



Key to the Display

Item		Description
(1)	Calibration curve lists	Displays a list of calibration curves for the selected
		measurement type.
(2)	Measurement type	Displays the measurement type selected with the function keys.
(3)	#	Calibration curve number. Highlighted if set only in the Conditions and measurement not implemented.
(4)	Conc.	Displays the concentration of the standards used to generate the calibration curve.
(5)	Amount	Displays the amount of the standard used to measure the calibration curve.
(6)	Status	 Displays the status of the calibration curve. The calibration curve cannot be deleted if either of the following two characters are displayed in this column. "S": calibration curve is being used for setting measurement conditions
		• "P": calibration curve is protected
(7)	Calibration curve data display	Displays the data for the selected calibration curve.
(8)	Graph	 A graph of the selected calibration curve is displayed here. It is automatically updated if the calibration curve is edited using the Curve Type or Curve Shift functions. Horizontal axis: Displays the amount of carbon. Vertical axis: Displays the area. Indicates each included measurement point.
(9)	Point display	 Displays the measurement results at each measurement point on the selected calibration curve. #: The number of each calibration point. Conc.: The standard concentration at the point. Amount: The amount of the standard. µgC: Carbon amount determined from carbon concentration and the weight or volume of the standard. Area: Area value for the measurement result at each point. #: Number of measurement repetitions.
(10)	R ²	Indicates the correlation coefficient (R^2) of the selected calibration curve. Displayed for a least squares method curve with at least three points.
(11)	Cell L	Indicates the length of the NDIR detector cell that was used.
(12)	Dens	Indicates the density of the standard.
(13)	Date	Indicates the date of the last revision.
(14)	Protect	Indicates the protection status of the calibration curve:OFF: not protectedON: protected

3.3 Measurements Using the TOC-VCSH/CSN/WS

3.3.8 Screen Displays

Item	Description
(15) Curve Type	Indicates the method by which the curve was created:
	Least squares: method of least squares
	• Pt to Pt: polygonal method
(16) Shift	Indicates whether the calibration curve is parallel shifted
	to pass through the origin (0 concentration point):
	none: not shifted to origin
	Origin: shifted to origin
Function Keys	
Reference mode	
Key	Function
F1 [Return]	Returns to the "Data Report - Measurement Log" screen.
F2 [TC], F3 [IC]	Switches the calibration curve measurement type.
F6 [Edit]	Enables editing mode for the selected calibration curve.
Editing mode	
Key	Function
F1 [Cancel]	Returns to the reference mode without making the changes.
F2 [Protect ON/OFF]	Use this to turn the protection function ON or OFF.
F3 [Cal. Curve Type]	Use this to select least squares display or Pt to Pt.
F4 [Zero Shift Status]	Select none or Origin.
F6 [OK]	Returns to the reference mode after making the changes.

3.3.8.5 "SSM Calibration - Settings"

Press the [Cal] key in the "Initial Display" to display the "SSM Calibration - Settings" screen.



Key to the Display

Item		Description
(1)	Screen title	"SSM Calibration - Settings".
(2)	Cal. Curves	Number of currently set calibration curves and maximum number of calibration curves that can be set.
(3)	Calibration curve information	 Displays information on previously set calibration curves for the selected measurement type. SSM-TC, SSM-IC: calibration curve type. #: calibration curve number.
		• Concentration: Maximum concentration of the stan- dard used to create the calibration curve.
		• Amount: Amount of the standard used to create the calibration curve.
		 Status: Status of the calibration curve. "S": Calibration curve is being used for sample
		measurement.
(4)	Calib. #	Calibration curve number (0 to 9). Enter a previously set calibration curve number to display setting details for that calibration curve
(5)	Amount Unit	Select whether the standard amount is entered as a mass or a volume.
(6)	Cell L	Displays which NDIR detector cell type is used.
(7)	Density	Enter the density of a liquid standard.
(8)	[***.***]	Defines the setting range of the item currently selected by the cursor.
(9)	POINT 1 to 10	Displays the point number. To add a calibration curve point, move the cursor to the solid box below the point number and press the right arrow key. To delete a calibration curve point, move the cursor to the solid box below the point number and press the F6 (Delete Point) key.
(10)	Conc	Enter the carbon concentration of the standard.
(11)	Amount	Enter the amount of the standard, as either a mass or volume.

Function Keys

Key	Function
F2 [SSM-TC]	Selects the SSM-TC calibration curve type.
F3 [SSM-IC]	Selects the SSM-IC calibration curve type.
F6 [Delete Point]	Deletes the selected calibration point.

Other Keys

Key	Function
[Cal]	Returns to the "Initial Display".
[Next]	Starts calibration curve measurement.

3.3.8.6 "SSM Calibration"

Press the [Next] key in the "SSM Calibration - Settings" screen to display the "SSM Calibration" screen.



Key to the Display

Item		Description		
(1)	Screen title	"SSM Calibration".		
(2)	Calib. #	Number of the calibration curve.		
(3)	Conc, Mean Area, SD, CV%	Displays the concentration and measurement results for each point.		
(4)	Peak graph	Displays a graph of the detector output during peak detection.		
(5)	Cnv Area	Area of nth measurement x (mass or volume of first measured sample/mass or volume of nth measured sample). SD and CV values in item ③ are calculated using the converted area.		
(6)	Remark	The following remark symbols are displayed.		
		• "T": Detector output tailing.		
		• "H": Detector output out of range.		
		• "F": Electric furnace temperature out of range.		
		"P": Incorrect sample boat position during measurement		
		"B". Unstable baseline during measurement		
(7)	Date & Time	Displays the current date and time		
(8)	Protect	Indicates the protection status of the calibration curve.		
		• OFF: not protected.		
		• ON: protected.		
(9)	Curve Type	Indicates the method by which the curve was created.		
		• Least squares: method of least squares.		
		• Pt to Pt: polygonal method.		
(10)	Shift	Indicates whether the calibration curve is parallel shifted to		
		pass through the origin (0 concentration point).		
		• none: Not shifted to origin.		
		• Origin: Shifted to origin (standard concentration 0 = area 0).		

Function Keys

Key	Function	
F1 [Add Injection]	Makes an additional measurement for the current point.	
F2 [Remeasure Point]	Repeats the measurement for the current point.	
F3 [Zoom In]	Enlarges the peak graph scale.	
F4 [Zoom Out]	Reduces the peak graph scale.	
F5 [Browse Cal. Curves]	Displays a graph of the carbon amount and area of the calibration curve.	
F6 [Exit Cal. Measurement]	Ends calibration curve measurement.	

3.3.8.7 "SSM Measurement Settings"



To start sample measurement, press the [Measure Sample] key in the "Initial Display". The "SSM Measurement Settings" screen is displayed.

Key to the Display

Item		Description
(1)	Sample name	Enter the sample name here.
(2)	[***.***]	Displays the setting range of the item currently selected
		by the cursor.
(3)	Calib.	Enter the number of the calibration curve to use for the
		measurement. Align the cursor with a measurement type
		in the calibration curve box to display the available
		calibration curves in the calibration curve list at the top
		of the screen. Enter the number of the desired calibration
		curve. The measurement range of each calibration curve
		is displayed to the right of the calibration curve number.
		Measurement is possible using a calibration curve in the
		list for which measurements are incomplete (calibration
		curve number is highlighted) but only the area result is
		displayed; the concentration is output as zero. The F1
		key is displayed as [Calibration Curve] when the cursor
		is in the calibration curve box.
(4)	Density	Enter the density of a liquid sample.
(5)	Cell Length	Displays which NDIR detector cell type is used.
(6)	Amount Unit	Sets whether the sample amount is entered as a mass or a volume. Press the [Select] key to toggle the setting.

Function Keys

Key	Function
F1 [Calibration Curve]	Opens the "Calibration Curve" screen. Except for the names of the function keys, this screen is identical to the "Data Report - Calibration Curves" screen. Select a calibration curve by pressing the cursor arrow keys to display detailed information for that calibration curve. Press the F6 [Select] function key in the "Calibration Curve" screen to select that calibration curve and enter it into the calibration curve box on the "SSM Measurement Settings" screen. Press the F1 [Cancel] key to return to the "SSM Measurement Settings" screen
F2 [SSM-TC], F3 [SSM-IC]	Selects the measurement type. Press the function key for the required measurement type to display the parameter box for that type and to select that measurement type for subsequent use. Press the same function key again to hide the parameter box and cancel the selection. The actual measurement is conducted left to right using each chosen measurement type.

3.3.8.8 "SSM Measurement"



Key to the Display

Item		Description	
(1)	Sample name	Displays the name of the currently measured sample.	
(2)	Area	Displays the mean area for multiple injections.	
(3)	Conc.	Displays the mean concentration for multiple injections.	
(4)	SD	Standard deviation of the concentration values.	
(5)	CV%	Coefficient of variance of the concentration values.	
(6)	TOC Conc.	The TOC value is automatically calculated if both TC and IC measurement types are selected.	
(7)	CNV	Displayed below a converted measurement item set on the "Conditions - Meas. Cond." screen. The character string "CNV" can also be changed to any other character string on the "Conditions - Meas. Cond." screen.	
(8)	Graph	Displays the peak waveform being measured.	
(9)	Remark	The following remark symbols are displayed:	
		• "T": Detector output tailing	
		• "H": Detector output out of range	
		• "F": Electric furnace temperature out of range	
		• "P": Incorrect sample boat position during measure- ment	
		• "B": Unstable baseline during measurement	
(10)	Date & Time	Displays the current date and time.	

Function Keys

Key	Function
F1 [Add Injection]	Makes an additional measurement.
F3 [Zoom In]	Enlarges the peak graph scale.
F4 [Zoom Out]	Reduces the peak graph scale.
F6 [Exit Cal. Measurement]	Ends measurement.

3.4 Measurement Using the TOC-VCPH/CPN/WP

This section describes the procedures used to perform measurements with the SSM-5000A when it is connected to the TOC-VCPH/CPN/WP.

3.4.1 Instrument Setup Wizard

The Instrument Setup Wizard is used to configure the SSM-5000A system.

The operations described here pertain only to the SSM-5000A.

For details on settings associated with the TOC-V, refer to Section 3.3 of the TOC-VCPH/CPN/WP manual.

System Setup Procedure

- *1*. Double-click the New System icon in the TOC-Control V main menu. The User dialog box is displayed.
- 2. Enter the user name and password. The Instrument Setup Wizard is displayed.
- **Reference:** For details on User Name and Password, refer to Section 3.2.1 of the TOC-VCPH/CPN/WP manual.
 - 3. Set the following parameters to create a system.
 - System Information

To use the SSM-5000A, enter a name for the System.

• Options

Oxidation Method: Select "Combustion".

Option: Select "SSM".

Instrument Setup Wizard	Options	×
	Ogidation : Combustion	
	Options: ♥10C ASI ♥SSM POC □IC Unit □TN □Sperge Kit □Manual Inj. Kit ■Pot Sampler-1 ♥	
	Define the attached options.	
	< Back Next > Cancel	

3.4.1 Instrument Setup Wizard

• TOC

Cell Length: Select "Short". Set the rest of the items according to the TOC-V system.

Instrument Setup Wizard TOC	×
Çətəlyst Type: <mark>Regular</mark> Iubing Diameter: Regular	- -
Ly Lemp Cell Length: Long Reactor Temp On Buzzer Auto regeneration of IC solution Egable ready status check TN Eower TN Eower	•
Specify the TOC parameters.	
< Back Next > Cancel	

• SSM

SSM TC Furnace Power On: Place a check in this box to heat the TC furnace when TC analyses will be conducted using the SSM-5000A.

SSM IC Furnace Power On: Place a check in this box to heat the IC furnace when IC analyses will be conducted using the SSM-5000A.

Instrument Setup Wizard	SSM X
	SSM IC Furnace On
	SSM IC Furnace Oni
	Specify SSM parameters
	< Back Next > Cancel
	-7

4. On the last page of the Instrument Setup Wizard, click Finish. A new system icon will be added to the TOC-Control V main menu.

3.4 Measurement Using the TOC-VCPH/CPN/WP 3.4.2 Setting Measurement Parameters

3.4.2 Setting Measurement Parameters

This section describes the procedure for setting the SSM-5000A measurement parameters.

↔SSM-500A Instrument Setup

Set the TC furnace and IC furnace temperatures as follows.

Procedure

- 1. In the TOC-Control V main menu, double-click the icon of the system to be used. The Instrument Properties dialog box is displayed.
- 2. Select the SSM tab.
- 3. Enter the parameters for the TC furnace and IC furnace.
 - Place a check in the SSM-TC Furnace Power On check box for TC measurement.
 - Place a check in the SSM-IC Furnace Power On check box for IC measurement.



3.4.3 Connecting the Instrument

3.4.3 Connecting the Instrument

This section describes the procedure for connecting the TOC-V and the personal computer.

....

Displaying Sample Table

- I. Double-click Sample Table in the TOC-Control V main menu.
- 2. Enter a user name and password in the User dialog box.
- **3.** To open a new sample table, select New from the File menu, or click the New button in the toolbar. A dialog box is displayed for selecting the type of file to be created.
- 4. Select the Sample Run icon, and click the OK button.



5. Select the system to be used from the drop-down list in the System tab of the General Information window.

General Informati	on
System History	
System:	SSM
Eile Version:	00.00.01E
<u>U</u> ser:	System
Date of Creation:	03/21/01 09:59:20 AM
<u>C</u> omment:	
OK	Cancel Apply

6. Click the OK button. A new sample table opens.

Connecting to the Instrument

Establish communication between the TOC-Control V software and the TOC-V as follows.

1. Select Connect from the Instrument menu, or click the connect button in the toolbar. The Sequence dialog box opens. Problems with the connection process are displayed here.

Sequence Sequence in prog Connect instrume	iress: ent			x
Task	Progress		Result	
Open Port TOC Initialize	100% now Initializing			
•				Þ
	Cancel]		

- 2. The Verify Parameters dialog box is displayed. Click on Connect Using New Settings.
 - *Note:* It takes 40 to 50 minutes for the furnace temperature to stabilize after turning the power On and establishing communication between the TOC-V and PC.

3.4.4 Calibration Curve Measurement

This section describes the procedure for performing calibration curve measurement.

3.4.4.1 Setting the Default Measurement Parameters

Default values are set for the following measurement parameters:

- Concentration Units
- Number of Injections

Procedure

- *1*. Select Default Measurement Parameters from the Options menu. The Default Measurement Parameters dialog box is displayed.
- 2. The following settings are made in the SSM-TC tab and the SSM-IC tab.
 - Units: Select the unit to be used for sample measurement.
 - Number of Injections: This is normally set to 1.

Default Measurement Parameters
TC IC NPOC POC TN SSM-TC SSM-IC
Units:
No. of injections:
OK Cancel Apply

3.4 Measurement Using the TOC-VCPH/CPN/WP

3.4.4 Calibration Curve Measurement

3.4.4.2 Creating a Sample Table

Use the following procedure to enter the sample table display items.

Procedure

- I. Select Display Settings from the Options menu.
- 2. Select Table Settings.
 - Set the appropriate items to be displayed in the sample table.

able Options		×
Sample Table Samples		1
Value 1 due Coulins. Value 2 due Coulins. Value 2 due 2 d	I Comment I Status I Action I Date / Time	
Select <u>A</u> ll	Select None Default	
OK	Cancel Apply	

.

3. To save the sample table, select Save As from the File menu. Enter a file name, and click Save.

Save As						? ×
Savejn:	🔄 Data		•	È	<u>r</u>	
🥦 Tutorial.t32	!					
File <u>n</u> ame:	SSM_1.t32					<u>S</u> ave
Save as <u>t</u> ype:	Sample Table:	s (*.t32)		•		Cancel
						<u>N</u> ew
Parameter		Value				
4						•

This saves the system and the measurement parameters. To use these parameters again, open this file and enter the parameters in the new sample table, and then save the file under a new name.

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3.4.4.3 Creating a Calibration Curve File

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The measurement parameters for generating a calibration curve are saved in a calibration curve file.

Procedure

1. Select New from the File menu, and then click the Calibration Curve icon.



2. Click OK to display the Calibration Curve Wizard (Page 1) System Information window.



3.4.4 Calibration Curve Measurement

- **3.** Select the analytical system from the System drop-down list, and click Next. The display proceeds to the Calibration Curve Wizard (Page 2) Calibration Curve Type window. Three types of calibration curves are available, as follows.
 - Calibration points are distributed uniformly over the calibration range: After entering the concentration range and number of calibration points, calibration point concentrations are automatically calculated by the software.
 - Edit calibration points manually: Concentrations are set manually by the user.
 - Calibration curve according to DIN38401/P-51: A calibration curve standardized on DIN (German standard).

anoration carro micara	(Page 2) Calibration Curve Type	×
Cal.Pts. Analysis Cal. Type System	Calibration points are distributed uniformly over the concentration range Edit calibration points manually Calibration curve according to <u>D</u> IN 38402/P-51	
	Calibration using automatic dilution C Div, Stendard Solutions / Fixed Dilution Dilution:	
Enter the calibration	C Fixed Standard Solution / Variable Dilutions	

4. Select the calibration curve type, and click Next.

The display proceeds to the Calibration Curve Wizard (Page 3) Analysis Information window. The following items are set in this page.

- Analysis: Select either SSM-TC or SSM-IC.
- Sample Name
- Sample ID
- Calculation method: Select either Linear Regression (Regression Linearity by Least Squares Method) or Point to Point.
- Zero Shift: Determines if the calibration curve is shifted to origin.
- File Name: Enter a name for the calibration curve file.

Calibration Curve Wizard (Page 3) Analysis Information	
Cal.Pts. 9Analysis •Cal. Type •System	Analysis: SSM-TC ▼ Default Sample Name: std Default Sample ID: 1 Calculation Method: Linear Regression ▼ ✓ Zero Shift ame: _TC_cal_1 	
Enter the ar	alysis parameters for the calibration curve.	
[< <u>B</u> ack Next > Cancel	

5. Set each of the items, and click Next.

The display proceeds to the Calibration Curve Wizard (Page 4) Calibration Point Parameters window. The following items are set in this page.

- Units: The Default Measurement Parameter value is automatically set here.
- Concentration: Concentration of the standard solution
- Number of Calibration Points: The number of calibration curve points (This is set only when "Calibration points are distributed uniformly..." is set in page 2.)
- Weight or Volume
- Density: Density is entered when the standard sample is a liquid.

Calibration Curve Wizard (Page 4) Calibration Point Parameter	×
Cal.Pts. Analysis Cal.Type System	Unit: [®] ▼ Concentration: 1.000 No. of Calibration Points: [™] [1-20] [™] Weight [®] Volume Volume Range: ³⁰ • ⁰ [ul Density: 1.000 [mg/ul] Cell Length: long	_
Enter corr	mon parameters for all calibration points]
	< <u>B</u> ack [<u>Next></u>] Cancel	

6. Set each of the items, and click Next. The display proceeds to the Calibration Curve Wizard (Page 5) Calibration Points List window.



7. Set or verify each of the parameters. To set a parameter, select the relevant row and click the Add button, or to change a setting, click the Edit button.

3.4 Measurement Using the TOC-VCPH/CPN/WP

3.4.4 Calibration Curve Measurement

8. Click Next.

The display proceeds to the Calibration Curve Wizard (Page 6) Peak Time Parameters window.

Calibration Curve Wizard (Page 6) Peak Time Parameters	X
Allowed peak start delay: DO DO THE DELAY	
Set the integration limits for the injections	
< <u>₿</u> ack <u>N</u> ext> Cancel	

9. Use this page to set whether the peak detection start should be delayed, or the peak detection time should be extended.

10. Click Next.

The display proceeds to the Calibration Curve Wizard (Page 7) History window.

Calibration Curve Wizard (F	Page 7) History	х
History	Enable history log User has to comment all modifications	
	Once the history function is enabled, it cannot be changed!!	
En	able / disable history functions	
	< <u>B</u> ack Finish Cancel	

The History log can be enabled here.

11. Click Finish.

The Calibration Curve Wizard closes, and the calibration curve file is saved.

3.4.4.4 Calibration Curve Measurement

Step 1 Setting up the Sample Table

- 1. Click the first row of the sample table, and select Insert. Then select Calibration Curve.
- 2. Select the file name of the desired calibration curve, and click the Open button. The calibration curve is automatically inserted into the sample table.

CONTROC-Control V Sample Tai	ble - [SS	SM_1.t32]						_ 8	x
Bile Edit ⊻iew Insert Inst	rument ,	Tools Signatures Options	: <u>W</u> indow					_ 8	×
D 😂 🖬 👗 🗅 🖻 e	S ?	🕺 🛛 🖻 🗞 🔿 🛢	: • • ×	B 2					
	Rdy:	N/A	Instr:	N/A	Mode:	N/A	Seq:	N	1/4
		Type Analysis	Sample Name	Sample ID	ObjectID	Origin	Dilution	Result	•
🕂 🗹 std	1	Standard SSM-TC	std	1	QA-000000-01	C:\Program Fil	1.000		
TC/TN System	3								
SSM	4								
	6								
	7								
	8								
	10								
	11								
	13								
	14								
	15								•
									_
×							-	$\ \times$	
								m 🚈	
								012	
								13 A	
							_	1 12 12	
Notification	s /(TC/	TN System λ SSM λ E	Errors ∖ Events	7.			Þ	1 2	
For Help, press F1						Syste	m	NUM	

Step 2 Measurement Procedure

1. Click Start in the Instrument Menu, or click the Start toolbar button. The TOC Measurement dialog box is displayed.



2.Click the Start button.

The Enter Sample Amount dialog box is displayed.

Enter San	nple An	oun	t	
Weight	0.000		mg	Start
Volume:	30		ul	Cancel
Density:	1.000		mg/uL	🔲 By Weight
Sample I	lame:	std		
Sample I	D:	1		
Analysis		SSM	1-TC	
Insert the on this	e sample dialog o	into t r on th	he SSM, ne instrum	and press [Start] ent front panel

- **3.** Transfer the standard to the sample boat. Enter the amount of standard placed in the sample boat.
- 4. Open the sample boat cover, and place the sample boat with its loaded standard on the sample boat transfer plate.
- 5. Securely close the sample boat cover.
 - **Note:** If the carbon content of the standard is 3.0mg or less (concentration x standard weight), wait 1.5 2 minutes after closing the cover before pressing the start button to avoid inclusion of an atmospheric CO_2 peak.
- 6. Verify the READY status of the instrument, and click the Start button. The message "Push the sample boat to the measurement position." is displayed on the screen.

SM Measurement				
Analysis:	SSM-TC			
Push sam	ple boat to the measurement position!			
	Close			

- 7. Push the sample boat into the furnace.
 - TC measurement: Gently push the sample boat transfer knob all the way to the MEASURING position (all the way to the back).
 - IC measurement: Operate the IC reaction solution (acid) injector to inject solution into the sample boat, and gently push the sample boat transfer knob all the way to the MEASURING position (all the way to the back).

Calibration curve measurement starts.

8. Click Sample Window in the View menu, or select the Sample Window button in the toolbar. The Sample Window is displayed to allow monitoring of the measurement in real time.

Step 3 Post Measurement Procedure

When sample measurement is completed, the display status changes as described below.

- TC measurement: The message "Pull the sample boat back to the COOLING position." is displayed.
- IC measurement: The message "Pull the sample boat back to the PREPARATION position." is displayed. Proceed to step 2 of this procedure.

SSM Measurement	
Analysis: SSM-TC	
Pull the sample boat back to the cooling position!	

 Return the sample boat transfer knob to the COOLING position. The sample boat transfer rod is cooled. After 30 seconds has elapsed, the message "Pull the sample boat back to the PREPARATION position." is displayed.

S	SM Mea	surement	
	Analysis:	SSM-TC	13
	Pull th	e sample boat ba preparation positi	ick to the on!
		Close	

- *Note:* Do not immediately return the TC furnace sample boat transfer knob to the CHANGE position from the MEASURING position, as this could cause damage.
- 2. Return the sample boat transfer knob to the CHANGE position.
- 3. The TOC Measurement dialog box is displayed.
 - *Note:* To perform additional measurements using the same standard, click the Repeat button. Repeat steps 2 and 3.

TOC Measur	ement				×
	_	SSM-TC			1
Spl. How.:	1	Inj. No.	Area	Conc.	Excluded
Det. No.:	1	1	536.8	0.000	
Inj. No.:	1				
Spl. Name.:	std		~~		
Spl. ID.:	1				
Mean Area:	536.8				
Mean Conc:	0.000%				
SD Area:	0.000	•			•
CV Area:	0.00	🗖 Fini	ished	Exc	lude
		Press (Rep	peat] to start continue the	another replical sample sequen	te or [Next] to ce!
		<u>R</u> epeat	,	Next	Stop

- <u>**TIP**</u> *»* To reject the measurement data, highlight the data to be excluded, and click the Exclude button.
 - To proceed to measurement at the next concentration (multi-point calibration curve): Press the Next button. Repeat steps 2 and 3.
 - To finish measurement: Click the Stop button.
 - *Note:* Occasionally, IC reaction acid overflows from the sample boat and accumulates on the IC sample boat holder. During sample changes, inspect and clean the sample boat holder as necessary.

3.4.5 Sample Measurement

This section describes the procedure for performing sample measurement.

3.4.5.1 Default Measurement Parameters

The following default parameters are entered for sample measurement.

- Concentration Units
- Number of Injections

Procedure

- 1. Select Default Measurement Parameters from the Options menu. The Default Measurement Parameters dialog box is displayed.
- 2. The following settings are made in the SSM-TC tab and the SSM-IC tab.
 - Units: Select the units to be used for sample measurement.
 - Number of Injections: This is normally set to 1.

Default Measurement Parameters	<
TC IC NPOC POC TN SSM-TC SSM-IC	
Units:	
No. of injections:	
OK Cancel Apply	

3.4.5.2 Creating a Sample Table

Set the sample table display items as follows.

Procedure

- *I*. Select Display Settings from the Options menu.
- 2. Select Table Settings.

Set the appropriate items to be displayed in the sample table.

Table Uptions	
Sample Table Samples	1
Type Analysis Sample Name Sample ID ObjectID ObjectID ObjectIn Ofligin Pollution Result Modified	Growment Status ØAction ØDate / Time
Select <u>A</u> ll	Select None Default
OK	Cancel Apply

3. To save the sample table, select Save As from the File menu. Enter a file name, and click Save.

Save As						?	х
Savejn:	🔄 Data		•	£	ď		
🕺 SSM_1.t32							1
🥦 Tutorial.t32							
File <u>n</u> ame:	sample_1.t32					<u>S</u> ave	
Save as type:	Sample Table:	s (*.t32)	_	•		Cancel	
						<u>N</u> ew	
Parameter		Value					
•							

4. This saves the system and the measurement parameters. To use these parameters again, open this file and enter the parameters in the new sample table, and then save the file under a new name.

3.4.5.3 Creating a Method File

The parameters to be used for sample measurement will be saved in a Method file.

Procedure

I. Select New from the File menu, and then click the Method icon.



2. Click OK to display the Method Wizard (Page 1) System Information window.

Method Wizard (Page 1)	System Informatio	n	×
Method Wizard (Page 1)	System Information System: Operator: Dete of Creation: Comment:	n System 03/21/01 10:20:51 AM	
Enter	the system informatic	n for the method	
< 8	ack <u>N</u> ext≻	Cancel	

- **3.** Select the analytical system to used from the System drop-down list, and click Next. The display proceeds to the Method Wizard (Page 2) SSM Analysis Information window. The following items are set here.
 - Analysis: Select either SSM-TC or SSM-IC.
 - Sample Name
 - Sample ID
 - Dilution: Dilution factor used during sample preparation.
 - Number of Determinations: Sets the number of times the same sample will be analyzed.
 - Density: Density is only entered for liquid samples.
 - Sample Amount Standard: Select weight or liquid, depending on whether the sample is a solid or a liquid.

• File name: Enter a name for the Method file.

Method Wizard (Page 2)	SSM Analysis Informat	ion 🗙
• Cal. Curve • Gen. Info. • System	Analysis: Default <u>S</u> ample Name: Default <u>S</u> ample <u>I</u> D: <u>D</u> ilution: <u>N</u> o. of Determinations: D <u>e</u> nsity:	SSM-TC Sample 1 1 000 1 0 1 00 1 0 1
Enter the analysis type, the	Her sample amount using: le Name: SSM_TC_met_1 e default sample name and method	Weight C Volume
< <u>B</u>	ack <u>N</u> ext>	Cancel

- 4. Set each of the items, and click Next. The display proceeds to the Method Wizard (Page 3) SSM Parameters window. The following parameters are set in this page.
 - Analysis: (read only)
 - Units: The Default Measurement Parameter value is displayed here.
 - Expected Concentration Range: Enter a value if the expected sample concentration is known.
 - Calibration Curve 1: Click the Browse button at the right of the entry field to select the calibration curve file, or enter the calibration curve file name directly.

Method Wizard (Page 3) SS	M Parameters		×
La Parameters	Analysis: SSN	I-TC E: Ci	xpected onc. Range
Cal. Curve	<u>U</u> nits: 🕅	• 1	.000
} → System			
	Calibration Curve <u>1</u> :		
	C:\Program Files\Shimad	zu Corporation	\TOC3
Define the	e injection parameters for th	e analysis	
< <u>B</u> ac	k <u>N</u> ext>	Cancel	

5. Set each of the items, and click Next. The display proceeds to the Method Wizard (Page 5) Peak Time Parameters window.

Method Wizard (Page 5) P	eak Time Parameters	x
	Analysis: SSM-TC	
	✓ Use gefault settings Allowed peak start delay: 00:00 20 10:00 20 10:00 20 14:00 20 14:00 20	
Set th	e integration limits for the injections	
< <u>B</u> a	ck <u>N</u> ext > Cancel	

- 6. Use this page to set whether the peak detection start should be delayed, or the peak detection time should be extended.
- 7. Click Next.

The display proceeds to the Method Wizard (Page 7) History window.

lethod ₩izard (Pa	age 7) History	X
History	Enable history log User has to comment all modifications	
	Once the history function is enabled, it cannot be changed!!!	
	Enable / disable history functions	
	< Back Finish Cancel	

The History log can be enabled here.

8. Click Finish.

The Method Wizard closes, and the method file is saved.

3.4.5.4 Sample Measurement

Step 1 Entering the Sample in the Sample Table

1. Click the row of the sample table into which the sample should be inserted, and select Insert. Then select Sample.

The Sample Wizard (Page 1) Parameter Source window is displayed.



- 2. Set the source of the measurement parameters (method or calibration curve) or if no source file is used, select "Edit parameters manually".
- **<u>TIP</u>** » *If multiple samples are to be measured using the same parameters, the settings can be made using the Insert Auto Generate function.*
 - 3. Click Next to display the Sample Wizard (Page 2) Analysis Information window.
- **TIP** » By selecting "Skip remaining Wizard pages and use measurement parameters from the source", the sample is inserted into the sample table using measurement parameters from the source file.
 - 4. Verify each of the parameters, and change settings as necessary.
 - 5. Click Next to display the Sample Wizard (Page 3) Calibration Curve window.
 - 6. Verify each of the parameters, and change settings as necessary.
 - 7. Click Next to display the Sample Wizard (Page 4) Injection Parameters window.
 - δ . Verify each of the parameters, and change settings as necessary.

9. Click Finish.

The sample is inserted into the sample table and displayed with all of the sample measurement parameters entered.

TOC-	Control	V Sample T	able - [san	ple 1.t32]					X
Eile	<u>E</u> dit <u>V</u> i	iew <u>I</u> nsert I <u>n</u>	strument <u>T</u>	ools <u>S</u> ignatures	<u>Options</u>	/indow		_ 8	Ľ
🗅 🖬	¥ 🖬 📗	ኤ 🖻 🛍	3	N? 🛛 🖂 ጷ	0 🖲 🖲		🖉 🗠 E	i 👪 👘	
	Rdy:	NZ	A,	Instr:	N/A	Mode:	N/A	Seq:	\square
		Туре	Analysis	Sample Name	Sample ID	ObjectID	Origin	Dilution	
	1	Unknown	SSM-TC	sample	1	QA-000000-01	C:\Program Fil	1.000	ō
	2								
	3								
	5			-					
	6								
	7								
	8							Þ	2
× 03/2 03/2 03/2		15:41AM Rd 15:41AM Ins 15:41AM Ins Notification	ly.Sis.Ready str Sis: Read str Mode: R is À TC/TI	iy eady N System ∑ \$	SSM / Erro	rs 💶 🕨			
For Help,	press F1					System		NUM	

↔ Step 2 Measurement Procedure

- 1. Click Start in the Instrument Menu, or click the Start toolbar button.
- 2. The Enter Sample Amount dialog box is displayed.

Enter Sar	nple Am	ounl		
Weight	100		mg	Start
Volume:	0		ul	Cancel
Density:	1.000		mg/uL	🔽 By Weight
Sample I	Name:	sam	ole	
Sample I	D:	1		
Analysis		SSM	I-TC	
Insert the	e sample dialog or	into t on th	he SSM, a he instrum	and press [Start] ent front panel

- **3.** Transfer the sample to the sample boat. Enter the amount of sample placed in the sample boat.
- 4. Open the sample boat cover, and place the sample boat with its loaded sample on the sample boat transfer plate.
- 5. Securely close the sample boat cover.
 - **Note:** If the carbon content of the sample is 3.0mg or less (concentration x sample weight), wait 1.5 2 minutes after closing the cover before pressing the start button to avoid inclusion of an atmospheric CO₂ peak.
- 6. Verify the READY status of the instrument, and click the Start button. The message "Push the sample boat to the measurement position." is displayed on the screen.

SSM Meas	SM Measurement			
Analysis:	SSM-TC			
Push sam	ple boat to the measurement position!			
,	Close			
- 7. Push the sample boat into the furnace.
 - TC measurement: Gently push the sample boat transfer knob all the way to the MEASURING position (all the way to the back).
 - IC measurement: Operate the IC reaction solution (acid) injector to inject solution into the sample boat, and gently push the sample boat transfer knob all the way to the MEASURING position (all the way to the back).

Sample measurement starts.

 δ . Click Sample Window in the View menu, or select the Sample Window button in the toolbar. The Sample Window is displayed to allow monitoring of the measurement in real time.

Step 3 Post Measurement Procedure

When sample measurement is complete, the display status changes as follows.

- TC measurement: The message "Pull the sample boat back to the COOLING position." is displayed.
- IC measurement: The message "Pull the sample boat back to the PREPARATION position." is displayed. Proceed to step 2 of this procedure.

SSM Meas	surement	
Analysis:	SSM-TC	
Pull the	e sample boat back to the cooling position!	

I. Return the sample boat transfer knob to the COOLING position.

The sample boat transfer rod is cooled. After 30 seconds has elapsed, the message "Pull the sample boat back to the PREPARATION position." is displayed.



- *Note:* Do not immediately return the TC furnace sample boat transfer knob to the CHANGE SAMPLE position from the MEASURING position, as this could cause damage.
- 2. Return the sample boat transfer knob to the CHANGE SAMPLE position.

3. The TOC Measurement dialog box is displayed.

To perform additional measurements using same sample, click the Repeat button. Repeat steps 2 and 3.

TOC Measur	ement						
		ľ	SSM-TC				
Spl. Row.: Det. No.: Inj. No.: Spl. Name.: Spl. ID.: Mean Area:	1 1 std 1 0.000		, <u>Inj. No.</u> 1	Area 0.000	Conc. 0.000	Exclude	ed
Mean Conc: SD Area: CV Area:	0.000% 0.000 0.00	-	Fini	shed		E <u>x</u> clude	•
			Press (Rep (<u>R</u> epeat	ieat) to star	t another rep e sample sec <u>N</u> ext	quence!	ktj to



- To proceed to measurement of the next sample: Press the Next button. Repeat steps 2 and 3.
- To finish measurement: Click the Stop button.
- *Note:* Occasionally, IC reaction acid overflows from the sample boat and accumulates on the IC sample boat holder. During sample changes, inspect and clean the sample boat holder as necessary.

3.4.6 Ending Measurement

This section describes the procedure for ending measurement.



1. Click Standby in the Instrument menu of the sample table. The Standby dialog box is displayed.



- 2. The following two methods are available for ending measurement operations. Select the appropriate method.
 - Shut down instrument: Used to switch off power to the instrument.
 - Auto Restart: Used to maintain the instrument in standby state where the furnace can be turned off and the carrier gas flow stopped. In this state the instrument can be set to automatically restart at a later date and time.

- Turn the power to the SSM-5000A TC furnace off at least 30 minutes before turning off the power to the instrument. If the instrument is switched off without allowing the TC furnace to cool, the cooling fan for the TC furnace stops operating, causing accelerated wear of the O-rings due to the high temperature inside the furnace. This procedure does not apply in emergency situations.
 - If the SSM-5000A is to remain idle for an extended period of time, flush out any phosphoric acid remaining in the tubing with water. If phosphoric acid is allowed to remain in the tubing, solid particles may form and create an obstruction inside the tubing.
 - Use a cotton swab or equivalent to remove acid or other corrosive substances that may come in contact with the inside of swage lock fittings or any of the TC and IC sample port parts, such as the sample boat holder or sample boat push rod. Wash the affected surfaces with water for any especially corrosive substances.

3.4.6.1 Shutdown

This section describes the procedure for switching off power to the instrument.

Procedure

- 1. Select Shut down instrument in the Standby dialog box, and click Standby. Power to the electric furnace is automatically turned off, the temperature decreases and the carrier gas flow stops. After 30 minutes elapses, the TOC-V power is automatically turned off.
- 2. Turn off the SSM-5000A and stop the flow of carrier gas to the SSM-5000A.

3.4.6.2 Auto Restart

This function allows the instrument to enter a standby state where the furnace is off and the carrier gas flow has been stopped. Approximately one hour before a predetermined time, the instrument automatically turns the flow of the carrier gas back on and turns the power on to the furnace. The instrument returns to a state of readiness at the predetermined time.

.....

Procedure

- I. Select Auto Restart in the Standby dialog box.
- 2. Select the desired instrument parameters (TC furnace, carrier gas, SSM TC furnace, SSM IC furnace), and set the startup date and time. Place a check in the box to the left of each of the units to ensure that the power to the respective unit will remain on and that the carrier gas will keep flowing. If the power is to remain off during the inactive period, do not place check marks in these boxes.
- 3. Click Standby.

The specified units will be turned off automatically. Approximately one hour prior to the set time, the power to each of those units will turn on automatically, allowing measurement to start at the set time.

<u>TIP</u> » To cancel the Standby setting, click Escape.

Note: The carrier gas ON/OFF setting refers only to the TOC-V carrier gas. Carrier gas supply to the SSM-5000A must be turned on and off manually.

3.4.7 Description of Sample Data

This section describes SSM-5000A-related data items in the TOC-Control V sample table. For details on other items, refer to the Sample Table Editor section of the TOC-VCPH/CPN/WP User Manual.

3.4.7.1 View Menu

ACalibration Curve

To view the calibration curve properties associated with a particular row in the sample table, click on that row and select the Calibration Curve command from either the View menu or the toolbar.

The Calibration Curve Properties dialog box opens.

Calibration Curve Properties	Þ
Common Parameter Analysis Data Graph History	
System: SSM	
User: System	
Date of Creation: 10:05:14 AM 03/21/01	
Comment:	
OK Cancel Apply	

Figure 3.8 • Common Tab of Calibration Curve Properties Window

Common Tab

Following is a description of calibration curve items in the Common tab of the Calibration Curve Properties window.

Setting Item	Description
System	The system used to measure the calibration curve is
	displayed in the drop-down list box.
User	This is a read-only field displaying the name of the user
	that generated the calibration curve.
Date of Creation	This is a read-only field displaying the date and time that
	the calibration curve was generated.
Comment	A text comment related to the calibration curve may be
	entered in this Comment field.

3.4 Measurement Using the TOC-VCPH/CPN/WP

3.4.7 Description of Sample Data

Parameter Tab

alibration Curve Prope	rties
Common Parameter A	halysis Data Graph History
<u>A</u> nalysis:	SSM-TC Status: Aborted
<u>U</u> nits:	× ×
Sample <u>N</u> ame:	Std
Sample <u>I</u> D:	1
No. of Cal. <u>P</u> oints:	2
<u>C</u> atalyst Type:	
Calibration Curve <u>Type</u> :	Standard Calibration Curve
Calculation Method:	Linear Regression 💌 🔽 Zero Shift
🔲 Multipl <u>e</u> Injections	
Calibration Curve <u>File</u> :	C:\Program Files\Shimadzu Corporation\TOC
	OK Cancel Apply

Figure 3.9 • Parameter Tab of Calibration Curve Properties Window

Setting Item	Description
Analysis	Displays the calibration curve measurement type.
Units	Displays the unit of concentration.
Sample Name	Enter a sample name using up to 64 characters.
Sample ID	Enter a sample ID using up to 64 characters.
Number of Cal. Points	Displays the number of calibration curve points.
Catalyst Type	Displays the catalyst type used during measurement.
Calibration Curve Type	Displays the type of calibration curve.
Calculation Method	Calculation Method sets the method of calculating the calibration curve. Two types of curves are available: Point-to-Point and Linear Regression. In the Point-to-Point type, a curve is drawn by connecting adjacent data points with a straight line. In the Linear Regression method, the line which best matches the pattern of the data points is determined statistically. The degree of agreement between the calculated line and the data points is called the correlation coefficient (r^2) . $r^2 = 1$ means that the line perfectly matches the data points. The general equation for a straight line is: Y=mX+b
	Here, $Y = Y$ axis data point
	X=X axis data point
	b= Y intercept
	Select Linear Regression or Point-to-Point from the drop-down list box.
Zero Shift	The Zero Shift option allows the calibration curve to be shifted so that it passes through the zero point. Note that the slope of the line remains unchanged. If the calibration curve is to pass through the origin, select Zero Shift.
File Calibration Curve	Displays the file name for the calibration curve.

Analysis Tab

The Analysis tab includes supplementary analysis parameters.

alibration Curve Properties	
Common Parameter Analysis Data Graph	n History
Analysis Parameter Density: 1.000 ug/ul © Vglume © Weight	Peak Time Parameters
	and Kardy

Figure 3.10 • Analysis Tab of Calibration Curve Properties Window

3.4 Measurement Using the TOC-VCPH/CPN/WP

3.4.7 Description of Sample Data

Analysis Parameters

Parameter	Description
Density	Displays the density when the sample is a liquid.
Volume or Weight	Displays either volume or weight depending on whether
	the standard is a liquid or solid, respectively.

Peak Time Parameters

Parameter	Description
Use default settings	This box is checked as the default. Remove this
	selection as necessary.
Allowed peak start delay	Indicates the time in minutes up to the time that
	measurement was stopped when a peak could not be
	detected. This option cannot be used if Use default
	settings is selected.
Total integration time	Indicates the time in minutes that measurement
	continues following detection of peak end. This option
	cannot be used if Use default settings is selected.

Data Tab

The Data tab displays information about each of the calibration points used to generate the calibration curve.

Calibration C	Curve Propert	ies				х
Common F	Parameter Ana Points:	alysis Data	Graph Hi	story		
No.	Conc.	Mean CNV	No. of Ini.	Excluded	Weight [mg]	
1	1.000 %		1/1		30.00	
2	0.000 %		1/1		30.00	
(3)						
•				J	Þ	
	Add.		lata	Dialata All	Fueluda	
Ealt	Baa	De	iere	Belete Bill	ERonne	
		ОК	Cancel	Δoc	in I	
			Caricer	<u>0</u> pp	<i>y</i>	



Column Heading	Description
No.	Calibration point number.
Concentration	Concentration of calibration point.
Average Conversion Value	Average conversion value.
No. of Injections	Number of injections.
Exclusion	"E" indicates that the calibration point has been
	excluded as a data value.
Weight	Weight of standard specimen.
Volume	Volume of standard solution.
Absolute Carbon Amount	Carbon amount determined from volume or weight of standard.

Column Heading	Description
SD	Standard Deviation.
CV	Coefficient of Variation.

A series of buttons is arranged below the data columns.

Button	Function
Edit	Used to edit the calibration point parameters. For details,
	refer to the description of the Calibration Curve Wizard
	in the TOC-VCPH/CPN/WP User Manual.
Add	Add a new calibration point. For details, refer to the
	description of the Calibration Curve Wizard in the
	TOC-VCPH/CPN/WPUser Manual.
Delete	Deletes the selected calibration point.
Delete All	Deletes all of the calibration points.
Exclude	Excludes the selected calibration point from the
	calculations.

Graph Tab

The Graph tab displays the graph of the calibration curve.



Figure 3.12 • Graph Tab of Calibration Curve Properties Window

Parameter	Description
Slope/Intercept/Correlation	These parameters display the value of the least squares
Coefficient	as explained in Section "3.4.7.1 View Menu" under the
	Parameter tab.

History Tab

If the History log was enabled when the calibration curve was created, the History tab displays information related to changes in the analysis history and parameters. If the History log was not enabled when the calibration curve was generated it can be enabled here. Once the History log is enabled, it cannot be disabled.

Calibration Curve Proper	ties	×
Common Parameter Ar	alysis Data Graph History	Edit Comment
Date/Time	User Changed Item	From
	OK Cancel Ap	

Figure 3.13 • History Tab of Calibration Curve Properties Window

Setting Item	Description
Enable History Log	If selected, the software will monitor all changes related
	to the system.
User has to comment all	This option is selected when comments and approvals
modifications	are required for all changes.
Edit Comment	The Edit Comment command displays the Comment
	dialog box. This dialog box is normally used to record
	the reason for modifying parameters or the method.
	When the User has to comment all modifications option
	is selected, the OK and Cancel buttons are disabled until
	a comment is entered to prevent continuing without
	recording a comment.

☆Method

To display the sample or method properties for a particular row in the sample table, click on that row, and select the Method command from the View menu or from the toolbar.

The Sample/Method Properties dialog box opens.

Sample / Method P	roperties 🛛 🗙
Common Paramete	SSM-TC History
System:	SSM
<u>U</u> ser:	System
Date of Creation:	03/21/01 10:20:51 AM
<u>C</u> omment:	
	OK Cancel Apply

Figure 3.14 • General Tab of Method Properties

General Tab

Setting Item	Description
System	Displays the system used for the measurement.
User	Display the name of the user who created the sample method.
Creation Date	Displays the date and time that the sample method was created.
Comment	Enter a comment consisting of up 512 characters.

3.4 Measurement Using the TOC-VCPH/CPN/WP

3.4.7 Description of Sample Data

Parameter Tab

Sample / Method Prope	rties X
Common Parameter SS	SM-TC History
<u>A</u> nalysis: Dilution: Sample <u>N</u> ame: Sample_ID: No. <u>o</u> f Determinations:	SSM-TC Status: Defined Sample 1 Use Weight C Use Volume
<u>D</u> ensity:	1.000 ug/ul
Current Method:	C:\Program Files\Shimadzu Corporation\TOC
	OK Cancel Apply

Figure 3.15 • Parameters Tab

Setting Item	Description
Analysis	Displays the analysis type (SSM-TC or SSM-IC or
	SSM-TOC).
Dilution	Enter the preparation dilution factor used in the
	concentration calculation. The default value is 1.0, and
	setting range is 0 - 100.
Sample Name	Enter a sample name using up to 64 characters.
Sample ID	Enter a sample ID using up to 64 characters.
Number of Determinations	Set the number of times the same sample is analyzed.
Use Weight / Volume	Select whether a weight standard or volume standard is
	used, which depends on whether the sample is a solid or
	liquid.
Density	Enter the density when the sample is a liquid.
Current Method	Displays the method file name.

SSM-TC Tab



Figure 3.16 • SSM-TC Tab

Setting Item	Description
Analysis Parameters	Displays the units, number of injections and expected
	concentration range. The units and expected
	concentration range can be changed.
Peak Time Parameters	If the default settings are used, the Use default settings option is selected. Allowed Peak Start Delay indicates
	the time in minutes up to the time that measurement was stopped when a peak could not be detected. This is a
	value from 0 - 20 minutes. Total Injection Time
	indicates the time in minutes that measurement
	continues following detection of peak end. This is a
	value from 14-20 minutes.
Calibration Curve 1	The Browse button can be used to access the Open
	dialog box, and a calibration curve file can be selected, or a calibration curve file name can be entered directly.

SSM-IC Tab

The SSM-IC Tab displays the same options as the SSM-TC tab.

History Tab

If the History log was enabled when the method was created, the History tab displays information related to changes in the analysis history and parameters. If the History log was not enabled when the method was generated it can be enabled here. Once the History log is enabled, it cannot be disabled.

Sample / Method Proper	rties	X
Common Parameter S9	SM.TC History	
Enable history log	and the second t	Edit <u>C</u> omment
🗖 🖄 ser has to co		User Event
Date/Time	User Changed Item	Erom To
•		Þ
	OK Cancel	Apply
F ¹		

Figure 3.17 • History Tab

Setting Item	Description	
Enable History Log	If selected, the software will monitor all changes related	
	to the system.	
User has to comment all	This option is selected when comments and approvals	
modifications	are required for all changes.	
Edit Comment	The Edit Comment command displays the Comment	
	dialog box. This dialog box is normally used to record	
	the reason for modifying parameters or the method.	
	When the User has to comment all modifications option	
	is selected, the OK and Cancel buttons are disabled until	
	a comment is entered to prevent continuing without	
	recording a comment.	

3.4.7.2 Instrument Menu

Select the Background Monitor command from the Instrument menu or the toolbar to display the instrument parameters.

The Background Monitor dialog box opens.

SSM Tab

Background I TOC S C SSM C SSM- C SSM-	Monitor SSM Power TC Furnace IC Furnace		
	x1 1000 600 400 -100 0 5	x50	20

Parameter	Description
SSM Power	Displays the power on/off status of the SSM-5000A.
SSM TC Furnace	Displays the TC furnace temperature status of the SSM-5000A.
SSM IC Furnace	Displays the IC furnace temperature status of the SSM-5000A.

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3.4 Measurement Using the TOC-VCPH/CPN/WP

3.4.7 Description of Sample Data

3.4.7.3 Maintenance Menu

This section describes the Maintenance menu options.

Mechanical Check

The operational condition of various instrument components can be checked using this item. Service personnel primarily use this function.

SSM Tab

lechanical Check	
TOC SSM	
Output	Input
SSM Power	SSM IC Furnace Control
Sample Change Position(TC)	SSM IC Furnace Control
Measurement Position(TC)	Cooling Fan Control
Sample Change Position(IC)	AUX- <u>1</u>
Measurement Position(IC)	AUX-2
SSM TC Fumace Ready	AUX-3
🗖 SSM IC Fumace Ready	□ AUX- <u>4</u>
L AUX	□ AUX- <u>5</u>
	OK

• Output

Displays the state of various control signals transferred between the TOC-V and SSM-5000A.

Item	Description
SSM Power	A check mark is displayed when the SSM-5000A power is switched ON.
Sample Change Position (TC)	A check mark is displayed when the SSM-5000A TC sample boat transfer knob is positioned at the SAMPLE CHANGE position.
Sample Measurement Position (TC)	A check mark is displayed when the SSM-5000A TC sample boat transfer knob is positioned at the MEASURING position.
Sample Change Position (IC)	A check mark is displayed when the SSM-5000A IC sample boat transfer knob is positioned at the SAMPLE CHANGE position.
Sample Measurement Position (IC)	A check mark is displayed when the SSM-5000A IC sample boat transfer knob is positioned at the MEASURING position.
SSM TC Furnace Ready	A check mark is displayed when the SSM-5000A TC furnace temperature is in the READY state.
SSM IC Furnace Ready	A check mark is displayed when the SSM-5000A IC furnace temperature is in the READY state.

• Input

The items listed below can be switched ON/OFF using this function. To switch the item ON, place a check in the corresponding check box.

Item	Description
SSM TC Furnace	SSM-5000A TC furnace power
SSM IC Furnace	SSM-5000A IC furnace power
Cooling Fan	SSM-5000A TC furnace forward cooling fan

3.4 Measurement Using the TOC-VCPH/CPN/WP

3.4.7 Description of Sample Data



Maintenance and service are extremely important to ensure proper instrument operation and to obtain stable, accurate data.

4.1 Inspection

This section describes the parts and components requiring inspection and replacement and describes inspection and replacement procedures.

4.2 Troubleshooting

This section describes error messages displayed by the SSM-5000A and appropriate troubleshooting procedures.

4.1 Inspection

4.1.1 TC Combustion Tube and TC Catalyst

To avoid burn injuries, wait for the TC combustion tube and TC catalyst to cool to room temperature before starting maintenance work.

The electric furnace temperature is indicated in the SSM-5000A display window.

TC Combustion Tube

When the inside surface of the combustion tube reacts with salts contained in the samples, it devitrifies, or becomes crystalline in texture and turns white. The tube can continue to be used in this condition until it breaks or cracks, causing gas leaks.

Measurement of samples containing components that erode the quartz tube when burned (such as fluorine or alkaline components), combined with the high TC furnace temperature up to 900°C, can shorten the service life of the TC combustion tube.

TC Catalyst

It is time to replace the TC catalyst when the following phenomena occur:

- Measured values (peak area values) for a standard sample are lower than previous values obtained using the same measurement parameters.
- · Measurement repeatability accuracy deteriorates.

When such phenomena occur, first check that the gas flow line is not leaking. If no leaking is discovered, replace the TC catalyst.

The efficiency of combustion oxidation of a solid sample varies considerably according to the sample type and organic content. When evaluating the performance of the TC catalyst, use a standard of the same type and characteristics as the actual samples.

Measuring various amounts of the sample and checking for a linear proportionality relationship is a method of inspecting the oxidation catalyst using the actual sample. Use a sample with evenly distributed carbon for this method to reduce errors during sampling.

Generally, when performance of an oxidation catalyst deteriorates, the effects are larger at higher carbon content levels, such that the linear proportionality relationship is no longer maintained.

4.1.2 Checking for Gas Leaks

One problem that can affect measured values when using the SSM-5000 is gas leakage in the gas flow lines. If problems with measurement sensitivity or repeatability occur, first check for gas leaks.

Gas leaks may occur in the following areas:

- (1) At the seal between the sample boat push rod and sample port block (A in "Figure 4.1
 Sealed Parts of the Sample Boat Push Rod").
- (2) At the seal between the sample port cover and the sample boat access opening on the sample port (B in "Figure 4.1 Sealed Parts of the Sample Boat Push Rod").
- (3) At the connection between the flange on the sample port block and the flange on the TC combustion tube or IC reaction tube.
- (4) At the drain tubing in the drain vessel on the right side of the SSM-5000A.

The area (1) described above is a seal on a part that moves during each measurement. The Teflon O-ring seal is a consumable item, and is the most likely location for gas leaks to occur.

☆ Checking for Gas Leaks

Checking at Locations (1), (2), and (3)

Apply a leak-check fluid or dilute soapy water to the seal, and check for bubbles.

A more thorough gas leak check can be performed by temporarily stopping the flow of carrier gas (such as by blocking the outlet side of the IC reaction tube with a finger) to increase the gas pressure. Do not stop the gas for more than five seconds. If stopped for too long, the gas pressure can increase to levels that are high enough to cause damage to the piping.

Checking at Location (4)

Visually check for bubbles.

One or two bubbles may leak out if the gas pressure is temporarily increased. This does not represent a problem during measurement.

The drain tubing has a sealed construction to prevent escape of carrier gas. However, gas may escape if the water level in the drain vessel drops.

The construction of the tip of the drain tubing inside the drain vessel is shown in "Figure 4.2 • Drain Separator".

The increase in gas pressure during sample combustion causes a temporary rise in the gas pressure inside the drain separator. The float inside the tube drops together with the water level to seal the opening of the ring at the tip of the tube, thereby preventing the carrier gas from escaping through the tip of the carrier gas discharge tubing.

Note: This construction is designed to temporarily prevent large amounts of gas from leaking; it cannot completely prevent gas leaks over long periods of time.

If the water level in the drain vessel drops, the float continuously seals the ring, preventing discharge of carrier gas or drain water.

Note: Supply enough water to the drain vessel to ensure the float is separated from the ring, as drain water cannot be discharged if the water level is too low.

The supply of drain water to the drain vessel during SSM-5000A measurement is less than during TOC-V measurement. Therefore, it is easy for the water level in the drain vessel to drop. Check the drain vessel water level before starting measurements.



(Float drops with water level to seal ring and prevent carrier gas leaks.)

Figure 4.2 • Drain Separator

4.1.3 Replacing Gas Seals

As shown in "Figure 4.3 • Replacing the O-Rings - 1", O-rings are used at the point where the sample boat push rod enters the sample port block.

Gas can leak around the O-rings due to wear, deformation, or scratching. Replace the O-rings when a leak is detected during the gas leak inspections and during periodic maintenance.

Also replace the O-ring used between the sample port block upper surface and the sample port cover, and the O-rings used in the TC combustion tube or IC reaction tube connectors if they leak gas due to deformation or scratching.

Reference: See Section "5.6.8.2 Installing the TC Combustion Tube and IC Reaction Tube".

Replacing O-Rings Between the Sample Port Block and Sample Boat Push Rod

Note: Turn off the SSM-5000A power before replacing the O-rings.

- *I*. Remove the front cover.
- 2. Holding the knurled part of the O-ring installation adapter, with the sample boat push rod inserted, turn it counterclockwise to remove it.
- **3.** Push the sample boat push rod knob about 4 to 5cm into the furnace from the SAMPLE CHANGE position.
- 4. Open the sample port cover, and fit the O-ring removing adapter over the sample boat push rod through the sample port opening.



Figure 4.3 • Replacing the O-Rings - 1

5. Pull back the sample boat push rod knob to the SAMPLE CHANGE position. The two O-rings can now be removed from the sample port block.

6. Loosen the Phillips screw at the rear of the sample boat push rod knob, and pull the sample boat push rod from the knob.



Figure 4.4 • Replacing the O-Rings - 2

- 7. Remove the O-ring installation adapter and two O-rings.
- δ . Verify that there is no foreign matter stuck to the surface of the sample boat push rod.
 - *Note:* Any foreign matter that is stuck to the surface of the sample boat push rod prevents the rod from sliding smoothly and may cause gas leaks due to accelerated wear or scratching of the Teflon O-rings.

If foreign matter is found on the surface of the sample boat push rod, clean it off or use a soft cloth to polish the surface of the rod.

If foreign matter is stuck to the part of the rod that enters the furnace and cannot be easily wiped off, wash it in approximately 1M hydrochloric acid or polish with polishing powder. Using polishing powder may reduce the diameter of the sample boat push rod, causing more frequent gas leaks.

- **TIP** » The surface of the TC sample boat push rod may become discolored due to the formation of an oxide film on the portion that is inserted into the furnace. This film does not interfere with analysis unless the surface of the sample boat push rod becomes roughened.
 - **9.** Reverse the procedure for removing the O-rings to put two new O-rings and the O-ring installation adapter on the sample boat push rod. Tighten the Phillips screw on the back of the sample boat push rod knob.
 - 10. Holding the knurled part of the O-ring installation adapter, turn it clockwise to screw it into the sample port block until the sample boat push rod resists movement.
 - II. Verify that no gas leaks from the Teflon O-rings.
- Reference: See Section "4.1.2 Checking for Gas Leaks".
 - 12. Apply a light coat of silicone grease (supplied with TOC-V) to the sample boat push rod on the IC side, so that it moves smoothly and easily.
 - *Note:* The IC sample boat push rod must be moved gently to avoid spilling the IC reaction acid out of the sample boat.
 - Never use grease on the TC sample boat push rod. The grease will burn when the sample boat is moved into the TC furnace, generating CO₂ and creating a high margin of error in the measured values.

4.2 Troubleshooting

4.2.1 Error Messages

4.2.1.1 Error Messages Associated with the TOC-VCSH/CSN/WS

The following messages may be displayed in the center of the TOC-V screen. Take the appropriate remedial action as described in the table below.

Message	Description	Remedial Action
WARNING : SSM TC	The temperature of the	Press any key and wait for the furnace to reach the
FURNACE Temp. Hit	SSM-5000A TC furnace is	set temperature. If the set temperature is not
Any Key.	not within the designated	attained, see Section "4.2.2.2 TC or IC Furnace
	range (900 $\pm 10^{\circ}$ C).	Does Not Reach Required Temperature".
WARNING : SSM IC	The temperature of the	Press any key and wait for the furnace to reach the
FURNACE Temp. Hit	SSM-5000A IC furnace is	set temperature. If the set temperature is not
Any Key.	not within the designated	attained, see Section "4.2.2.2 TC or IC Furnace
	range (200 $\pm 10^{\circ}$ C).	Does Not Reach Required Temperature".
WARNING : SSM	The SSM unit power is not	Press any key. Ensure that the SSM-5000A power
Disconnected Hit Any	connected correctly.	supply is correctly connected. See Section "4.2.2.1
Key.		SSM-5000A Does Not Operate".

Table 4.1 •	TOC-V	VCSH/	CSN/WS	Error	List	1
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Table 4.2 • TOC-VCSH/CSN/WS Error List 2

Message	Description	Remedial Action
SSM poweris OFF.; Turn it on and press any key.	The SSM power is not turned on.	Press any key. Turn on the SSM-5000A power. See Section "4.2.2.1 SSM-5000A Does Not Operate"
TC boat is in the MEA- SUREMENT position. Pull it back to the SAMPLE CHANGE position and press any key.	The TC sample boat was not in the SAMPLE CHANGE position when the [Start] key was pressed.	Press any key. Move the TC sample boat to the SAMPLE CHANGE position.
IC boat is in the MEA- SUREMENT position. Pull it back to the SAMPLE CHANGE position and press any key.	The IC sample boat was not in the SAMPLE CHANGE position when the [Start] key was pressed.	Press any key. Move the IC sample boat to the SAMPLE CHANGE position.

The following messages may be displayed in the center of the TOC-V screen. The red warning indicator simultaneously flashes. These messages indicate that a fatal error occurred and operation cannot continue.

Message	Description	Remedial Action
Error!! SSM TC Boat	The TC sample boat posi-	Turn off the instrument. Contact your Shimadzu
Position Sensor	tion detector photosensors	representative.
	(two locations) are not	
	working properly. The light	
	path is obstructed for both	
	photosensors at the same	
	time.	
Error!! SSM IC Boat	The IC sample boat posi-	Turn off the instrument. Contact your Shimadzu
Position Sensor	tion detector photosensors	representative.
	(two locations) are not	
	working properly. The light	
	path is obstructed for both	
	photosensors at the same	
	time.	

Table 4.3 • TOC-VCSH/CSN/WS Error List 3

\cancel{P} Displaying and Clearing the Error Log

When an error message in Lists 1 or 3 is displayed, the date and time the error occurred and the error item are displayed in the Error Log.

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Displaying the Error Log

- *I*. Press the F5 [Maintenance] key on the "Initial Display". The "Maintenance" screen is displayed.
- 2. Press the F6 [Error Status] key on the "Maintenance" screen to display the Error Log.

Clearing the Error Log

Press the F3 [Clear Error Log] function key to clear all entries in the Error Log.

4.2.1.2 Error Messages Associated with the TOC-VCPH/CPN/WP

The following messages are displayed when an error is detected during parameter entry or operation. Use the remedial measures listed in the table below to alleviate the situation.

Message	Description	Remedial Measure
ERROR: SSM TC Fur-	The SSM-5000A TC fur-	Wait until the temperature reaches the set temper-
nace	nace temperature is not	ature. If it does not reach the set temperature, refer
	within the setting range	to Section "4.2.2.2 TC or IC Furnace Does Not
	$(900 \pm 10^{\circ} \text{C}).$	Reach Required Temperature".
ERROR: SSM IC Fur-	The SSM-5000A IC fur-	Wait until the temperature reaches the set temper-
nace	nace temperature is not	ature. If it does not reach the set temperature, refer
	within the setting range	to Section "4.2.2.2 TC or IC Furnace Does Not
	$(200 \pm 10^{\circ} \text{C}).$	Reach Required Temperature".
ERROR: SSM Con-	The SSM-5000A is not cor-	Verify that the SSM-5000A is correctly con-
nection	rectly connected.	nected. Refer to Section "4.2.2.1 SSM-5000A
		Does Not Operate"

Table 4.4 • TOC-VCPH/CPN/WP Error Message List 1

The following messages indicate that an error has occurred that prevents measurement operations from continuing. Take appropriate measures as indicated in the table.

Table 4.5 • TOC-VCPH/CPN/WP Error Message List 2

Message	Description	Remedial Measure
ERROR: SSM TC	The TC sample boat position detector	Turn off the instrument. Contact your
sample boat position	photosensors (2 locations) are not	Shimadzu representative.
	working properly. The light path is	
	obstructed for both photosensors at	
	the same time.	
ERROR: SSM IC sam-	The IC sample boat position detector	Turn off the instrument. Contact your
ple boat position	photosensors (2 locations) are not	Shimadzu representative.
	working properly. The light path is	
	obstructed for both photosensors at	
	the same time.	

4.2.2 Troubleshooting Flowcharts

This section describes relatively simple inspection and repair procedures that can be performed by the user.

Use these flowcharts in conjunction with the appropriate "Troubleshooting" flow charts in the associated TOC-V User Manual.

Contact your Shimadzu representative if the malfunction cannot be remedied by the recommended procedures or if the replacement of parts is required.

4.2.2.1 SSM-5000A Does Not Operate





4.2.2.2 TC or IC Furnace Does Not Reach Required Temperature



4.2.2.3 No Peaks Generated During Standard Measurement



4.2.2.4 Repeatability is Poor Even for Satandard Measurement

4.2 Troubleshooting

4.2.2 Troubleshooting Flowcharts

Reference Information

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5

This chapter presents technical information, such as the specifications, accessories, and installation procedures in the event that the instrument is moved.

5.1	Specifications
	The SSM-5000A specifications are listed here.

- 5.2 Accessories List This section contains the accessories list for the SSM-5000A.
- 5.3 Special Accessories List This section contains the special accessories list for the SSM-5000A.
- 5.4 Consumables List The SSM-5000A consumables are listed here.
- 5.5 Service Parts List

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The SSM-5000A service parts are listed here.

5.6 Installation

This section describes the site conditions and installation procedures for the SSM-5000A.

5.1 Specifications

Measurement items	TC, IC, TOC (TC-IC)
TC oxidation method	Combustion oxidation (TC furnace temperature: 900°C, max. 980°C)
IC reaction method	Acidification (IC furnace temperature: 200°C)
*Measurement range	TC 0.1 to 30mgC, IC 0.1 to 20mgC
*Maximum sample weight	1g (up to 0.5g and 0.3g water content for TC and IC measurement, respec-
	tively)
*Repeatability Standard	±1% of range value
deviation	
*Measurement time	Normally, 5 to 6min (max. approximately 8min)
Carrier gas	Oxygen (99.9%), 500mL/min
Ambient temperature	5 to 35°C
Power requirements	100 to 120 V or 220 to 240 V (±10%) 7A, 50/60Hz
(SSM-5000A)	
External dimensions	Approx. 450mm (W) x 656mm (D) x 290mm (H) (excluding protruding
(SSM-5000A)	parts)
Weight (SSM-5000A)	Approx. 30kg

* These values will vary with sample type and analysis conditions.

5.2 Accessories List

Part No.	Part Name	Quantity	Remarks
638-93102-31	SSM-5000A Main Unit ASSY	1	For AC 100 to 115 V
638-93102-32	SSM-5000A Main Unit ASSY	1	For AC220 to 240 V
638-93103-31	Standard Accessory Set	1	For AC 100 to 115 V
638-93103-32	Standard Accessory Set	1	For AC220 to 240 V
630-02679	Sample boat	10	See Section 3.1.1
630-00566	Cobalt oxide catalyst	2	See Section 5.6.8
017-42801-01	Platinum catalyst	1	See Section 5.6.8
630-00557	Quartz wool	1	See Section 5.6.8
638-58102	Metal screen for catalyst	2	See Section 5.6.8
638-41325-01	TC combustion tube	2	See Section 5.6.8
638-41325-02	IC reaction tube	2	See Section 5.6.8
036-11404	O-ring Teflon P6	4	See Section 4.1.3
036-11237	O-ring, 4DP35	4	See Section 4.1.3
086-16101	Tweezers	1	See Section 3.1.1
071-60813	KPR-18 adapter	1	See Section 5.6.5
071-60814-01	AC power cord (for AC 100-115V)	1	See Section 5.6.5
071-60814-06	AC power cord (for AC 220-240V)	1	See Section 5.6.5
638-60074	Ceramic fiber	1	See Section 3.1.1
631-40284	Sample port adapter	2	See Section 5.6.7
036-11437	O-ring Teflon P35	2	See Section 5.6.7
036-11243	O-ring 4DP40	2	See Section 5.6.7
631-40273-01	Reducing elbow 6x4	1	See Section 5.6.6
086-03818	Wrench HX 1.5	1	See Section 4.1.3
631-41970	O-ring removing adapter	1	See Section 4.1.3
035-65413-01	T union	1	See Section 5.6.6
638-72559	Digital I/O board	1	See Section 5.6.3
638-74038	Cable	1	See Section 5.6.3
046-00199-06	Acid dispenser, DP-1-PTFE	1	See Section 5.6.9
631-46562	Tubing adapter	1	See Section 5.6.9
630-00315-09	Tygon tubing 9 ID x 12 OD	3m	See Section 5.6.10
037-61107-12	Hose clamp	1	See Section 5.6.10
016-37503	Teflon tubing 2 ID x 3 OD	1m	See Section 5.6.7
631-40193-01	Reducing connector	4	See Section 5.6.7
037-61107-06	Hose clamp	8	See Section 5.6.7
M393-E198	User Manual	1	

5.3 Special Accessories List

Part No.	Part Name	Remarks
630-08585-05	Cylinder pressure regulator	
638-56093	Cell switching valve set	Uses TOC-V 200mm cell, for high sensitivity measurement

5.4 Consumables List

Part No.	Part Name	Remarks
638-92099	Sample boat set	100 sample boats, includ-
		ing the ceramic fiber
630-00566	Cobalt oxide catalyst	
017-42801-01	Platinum catalyst	
630-00557	Quartz wool	
638-58102	Metal screen for catalyst	
638-41325-01	TC combustion tube	
638-41325-02	IC reaction tube	
036-11404-84	O-ring, Teflon P6	5 pieces included
036-11237-84	O-ring, 4D P35	5 pieces included
036-11243-84	O-ring, 4D P40	5 pieces included
638-60074	Ceramic fiber	

5.5 Service Parts List

Part No.	Part Name	Remarks
638-59143-01	Fan SSM ASSY.	-01, CN53 (condensation coil)
-02		-02, CN54 (TC furnace)
-03		-03, CN55 (TC combustion tube)
080-80123-04	T regulator, E5CS-Q1KJ	Temperature controller
638-65259-01	Thermocouple ASSY.	For TC furnace
638-65259-02	Thermocouple ASSY.	For IC furnace
074-80626-12	Power supply, WRA21FWX-B-U	Switching regulator
065-60036-08	Relay, G4W 11123A-DC24	
065-68001-05	Relay, G3NE 210TDC12V	SSR
638-65383	Tandem cell (in case)	
5.6 Installation

5.6.1 Before Installation

Verify Shipment

Confirm that all of the items listed in Section "5.2 Accessories List" are included.

5.6.2 Installation Site

Select an appropriate installation site, considering the instrument size, operation, and site conditions. Poor conditions at the installation site can cause errors in the results and damage to the instrument.

5.6.2.1 Selecting an Installation Site

The SSM-5000A is positioned to the right of the TOC-V, allowing the operator to use the keypad while viewing the LCD display of the TOC-V.



450mm

90

mm

440mm

Figure 5.1 • TOC-V + SSM-5000A External Dimensions (Example: TOC-VCSH)



- Internal parts reach very high temperatures and could cause a fire hazard. For safety and ease of maintenance, leave a space of at least 200mm between walls and the left, right, and rear of the instrument.
- Lift the SSM-5000A at the bottom of each side of the instrument and lift it by the base plate. Do not lift it by the front access door, as this may cause instrument damage.

656mm

5.6.2.2 Site Conditions

Select an installation site that complies with the following conditions:

- · A clean environment free of corrosive gases, organic gases, and dusts
- 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
- *Note:* A contaminated atmosphere can increase the measurement error, especially with high-sensitivity analysis.



Avoid locations where flames are prohibited. Some internal parts reach very high temperatures and could cause a fire hazard.

Due to the possibility of analysis error, avoid the following types of sites:

- Areas near heat sources
- · Areas next to windows receiving direct sunlight
- · Areas in direct exposure to air expelled from an air conditioner
- Areas near equipment generating strong magnetic fields, electric fields, or high frequencies

5.6.3 Electrical Connections

This section describes the electrical connections between the SSM-5000A and TOC-V.



Before proceeding with these operations, verify that the power to the TOC-V and the SSM-5000A is OFF.

Installing the Digital I/0 Board in TOC-V

- **1.** On the Digital I/O board, for SW1, set number 1 to ON and numbers 2, 3 and 4 to OFF. For SW2, either ON or OFF is acceptable. For JP1, place all of the jumpers between the center pin and the pin that is closest to the edge of the Digital I/O board.
- 2. Remove the screws on one of the option board installation rack cover plates on the rear panel of the TOC-V.

Note: Any of the three plates can be removed.

3. Insert the supplied digital I/O board into the option board installation rack.

Connecting the Cable

1. Identify the "TOC" and "SSM" indications on the ends of the provided connection cable and connect the cable between the TOC-V and the SSM-5000A, matching the indications with the appropriate unit.



5.6.4 Changing the Power Source Voltage

The SSM-5000A was set up when it was shipped to match the power source voltage of the region where the instrument is to be used. Contact your Shimadzu Service Representative if it is necessary to change the power source voltage after delivery. Depending on the voltage change, an authorized Shimadzu service engineer may be required to change wiring connections to the power supply, switch the terminal board, and/or make changes on the main board.

5.6.5 Power Supply and Ground Connection

Power Supply Connection

Connect the instrument to a 100 to 115VAC (100V series) or a 220 to 240VAC (200V series), 50/60Hz power supply with at least 7A capacity. The ~ symbol indicates an AC supply is required.



The instrument may not function correctly if the supply voltage does not meet or exceeds the 100 to 115VAC or 220 to 240VAC ranges. Unstable voltage supply may impede high-sensitivity measurements.

Note: This instrument can function at a 50Hz or 60Hz power supply frequency.

Ground Connection

The supplied power cable contains three leads, including a ground lead. The attached plug is a grounded, 3-pin plug. If the power socket is ungrounded, use the KPR-18 two-pin adapter. The ground pin must be connected to an external ground lead.

The live (AC) and ground (ACC) pins of the plug are arranged as shown in "Figure 5.3 • Electrical Plug". Connect it to a socket with the same configuration.



Figure 5.3 • Electrical Plug



- In addition to the safety considerations, complete grounding is extremely important to prevent disturbance noise from getting into the logic circuits and microcomputers. A Type-3 ground (ground resistance: 100W or less) is recommended.
- Perform grounding separately and avoid contact with water supply pipes, gas pipes, and lightning rods.

5.6.6 Connecting the Gas Tubing

5.6.6.1 Carrier Gas

Oxygen is used as the SSM-5000A carrier gas (and support gas).

Oxygen provides a higher combustion oxidation rate for high-carbon content solid samples. These samples have a higher carbon content and a lower contact efficiency between the sample and the carrier gas than the water/solution samples oxidized in the TOC-V.

Oxygen is used as the carrier gas to burn the sample in an oxygen-rich environment to achieve a high oxidation rate. Use oxygen gas of at least 99.9% purity.

Note: To perform high-sensitivity measurements using the TOC-V while it is connected to the SSM-5000A and is using oxygen as the carrier gas, the oxygen must be ultra pure. Using 99.9% oxygen in this situation may limit accurate analysis by generating larger blank peaks or false peaks during TOC-V measurements.

5.6.6.2 Gas Connections

Connection Procedure

- 1. Connect the oxygen gas cylinder to the gas inlet (GAS INLET) on the back of the SSM-5000A.
 - *Note:* Maintain a supply pressure of 300kPa (44PSI).
- <u>**TIP**</u> *w* Use of the air tubing set, available as a special accessory, is recommended. The air tubing set contains nylon tubing and connectors $(2 \times half-unions)$.
 - For connection to an oxygen gas cylinder (99.9%), connect a half-union to the cylinder pressure regulator, then connect the cylinder to the SSM-5000A using nylon tubing (4mm OD, 2.5mm ID).
 - When using a high-purity oxygen cylinder for both TOC-V and SSM-5000A, insert the T-shaped quick connector (SSM-5000A standard accessory) into the nylon tubing between the high purity gas cylinder and the TOC-V to create a connection for the SSM-5000A.
- **TIP** » Connect the nylon tubing to the half-union by pushing the tubing into the half-union. To disconnect the tubing, pull it out while firmly pressing the blue ring with a flathead screwdriver.
 - 2. Guide the Teflon tubing from the gas outlet (GAS OUTLET) on the SSM-5000A rear panel through the grommet in the upper part of the TOC-V rear panel.



Figure 5.4 • Flow Path of the SSM-5000A Flow Controller

5.6.7 Gas Connections to Cells

When the SSM-5000A is connected to the TOC-V, the gas tubing connections at the detector must be changed. The gas tubing from the TOC-V is connected to the long cell, and the gas tubing from the SSM-5000A is connected to the short cell.

Use the provided L-shaped reducing union to connect the Teflon tubing from the SSM-5000A gas outlet to the short cell inlet.

Attach the soft tubing that was between the long cell and the CO_2 absorber to the short cell outlet, and lead the other end out of the TOC-V.



Connect the provided Teflon tubing (3mm O.D, 2mm I.D.) between the long cell gas outlet and the CO₂ absorber gas inlet.

Figure 5.5 • Gas Connections to Cell

5.6.8 Installing the TC Combustion Tube and IC Reaction Tube



- To avoid burn injuries, allow the TC combustion tube and TC catalyst to cool to room temperature before performing maintenance. The electric furnace temperature is indicated in the SSM-5000A display window.
- The TC combustion tube is made of quartz glass and the IC reaction tube is made of hardened glass. Handle with care to avoid breaking and to prevent injury from broken glass.

5.6.8.1 Filling the TC Combustion Tube with Catalyst

The TC combustion tube is filled with the catalyst provided, as shown in "Figure 5.6 • Filling the TC Combustion Tube with Catalyst".

Procedure

- *I*. Prepare the catalyst mixture.
 - 2 containers of cobalt oxide catalyst (25g each).
 - 1 container of platinum catalyst (20g).
- 2. In a clean container, mix the catalysts to as uniform a mixture as possible.
- 3. Introduce approximately 45g of the catalyst mixture into the TC combustion tube.

- *Note:* Do not allow pulverized catalyst dust to be transferred into the TC combustion tube. This dust will increase resistance to the airflow through the TC combustion tube, making measurement impossible.
- 4. Fold the catalyst support screens as shown in "Figure 5.6 Filling the TC Combustion Tube with Catalyst", and insert them into the TC combustion tube.
- 5. Push the catalyst support screens into place with the catalyst filling rod supplied as a TOC-V accessory.
- **TIP** » Use the catalyst packing rod to pack the catalyst in a manner that creates some resistance between the inside of the combustion tube and the outside of the mesh. This prevents the mixed catalyst from crumbling and falling through the mesh and possibly flowing upstream toward the detector.

<u>TIP</u> *»* To prevent clustering of either type of catalyst, rotate the TC combustion tube while filling it with the catalyst mixture.



5.6.8.2 Installing the TC Combustion Tube and IC Reaction Tube



- To avoid burn injuries, allow the TC combustion tube and TC catalyst to cool to room temperature before performing maintenance. The electric furnace temperature is indicated in the SSM-5000A display window.
- Do not use wrenches or other tools as they may break the combustion tube and cause injuries.

Removing Front and Rear Covers



- 2. Move the sample boat push rod knob to the MEASURING position.
- 3. With the sample port cover closed, slightly lift the rear of the front cover, pull it about 8cm forward and lift it off.

Installing the O-Ring

- *I*. Remove the screws (4 knurled, 2 Phillips) then lift out the sample boat moving units.
- 2. Place the white Teflon O-ring P35 onto the TC combustion tube or IC reaction tube. Place the sample port adapter over the white O-ring as shown in "Figure 5.8 • Installing the O-Ring". Gently insert the tapered end of the combustion or reaction tube into the appropriate furnace.





- **3.** Insert the O-ring 4D P35 into the groove in the sample port block, as shown in "Figure 5.9 Installing the TC Combustion Tube or the IC Reaction Tube".
- 4. Return the sample boat moving unit to its original position.

Note: Pull the sample boat push rod knob forward to attach the two Phillips screws to the front.

5. Align the flange of the combustion or reaction tube and push the tube in while securing the sample port adapter to the sample port block. Sample Port Block – – O-Ring 4D P35



Figure 5.9 • Installing the TC Combustion Tube or the IC Reaction Tube

A Installing the TC Combustion Tube

Connect the glass L-pipe to the outlet side of the TC combustion tube (small diameter part) with the L-shaped swage lock union, as shown in "Figure 5.10 • Connecting the TC Combustion Tube to the Swage Lock Union".



Figure 5.10 • Connecting the TC Combustion Tube to the Swage Lock Union

Procedure

- 1. Loosen the screws that hold the rear access panel to the TC furnace in place. Lift up to remove the rear access panel.
- 2. Insert the tip of the combustion tube into the swage lock union. Lightly hand-tighten the box nut.
 - *Note:* With the box nut slightly tightened but the small diameter part of the tube still able to slide, slide the tip of the tube to create a clearance of 1 to 2mm from the contact face of the union.

3. Hand-tighten the box nut.

Sufficient sealing is obtained if the swage lock union does not move when pulled upward.

- Avoid using wrenches to tighten the box nut. The TC combustion tube is made of quartz glass, which will break if the box nut is over-tightened.
- The tip of the TC combustion tube may chip if the box nut is tightened with the tip of the tube touching the contact face of the union.
- Use only the supplied Teflon front and rear ferrules. Never use metal or other ferrules.
- Protect the inner and outer surfaces of the TC combustion tube tip, inner surface of the swage lock fitting, ferrules and other gas contact surfaces from grease, oils or other organic substances.

A Installing the IC Combustion Tube

Connect the outlet end of the IC reaction tube to a 4mm OD Teflon tubing with the reducing union for 6mm OD to 4mm OD.

5.6.9 Connecting the IC Reaction Acid Dispenser

Assemble the acid dispenser according to the following instructions, and position it to the right of the SSM-5000A.

Procedure

- I. Remove the Teflon elbow from the outlet of the acid dispenser.
- 2. Locate the acid injection tubing which is attached to the SSM-5000A IC sample port cover. Fully insert the free end into the tubing adapter (located in the SSM assessors packet).
- 3. Firmly push the tubing adapter into the acid dispenser outlet.



- 4. Fill the acid dispenser with water.
- 5. Place an empty sample boat into the IC sample port, and close the IC sample port cover. Operate the acid dispenser to confirm that the water is properly injected into the sample boat and that no water leaks from the tubing connections.

- 6. Replace the water in the acid dispenser with the supplied phosphoric acid. See Section "3.1.7 IC Reaction Acid" for details on the phosphoric acid concentration.
- 7. Operate the acid dispenser to purge the water from the tubing with the phosphoric acid.

TIP » The threads on the neck of the supplied glass container for phosphoric acid match those on the acid dispenser. This allows the acid dispenser to be attached directly to the glass phosphoric acid container.



- Phosphoric acid is highly corrosive. Use care to avoid touching or spilling it during handling. See the Material Safety Data Sheet (MSDS) section in the TOC-V User Manual for details.
- If phosphoric acid spills on any sliding parts, wipe it off immediately using a damp cloth.
- *Note:* The IC sample port cover should always remain in the closed position, except during sample transfer. Corrosion may occur if the sample port cover is left open and acid leaks onto the sliding mechanism of the sample port cover. Keep an empty sample boat in the IC sample port when it is not in use to catch any acid that may leak from the acid dispenser.

5.6.10 Drain Tubing

Connect the drain tubing $(12\text{mm OD} \times 9\text{mm ID}, \text{length }3\text{m})$ to the drain nipple on the back of the SSM-5000A, and direct it into an appropriate drain container. Connect the drain tubing tightly with the supplied hose clamp to prevent leaks.

<u>Reference:</u> For details about the drain tubing see the "Connecting the Drain Tubing" section in the TOC-V User Manual.



The drain liquid may contain acid or other corrosive substances. Use care to avoid touching or spilling the drain liquid.

5.6.11 Supplying Water to the Drain Vessel

Use distilled water or ion-exchange water to fill the drain vessel located on the right side of the SSM-5000A to the level of the drain outlet.

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