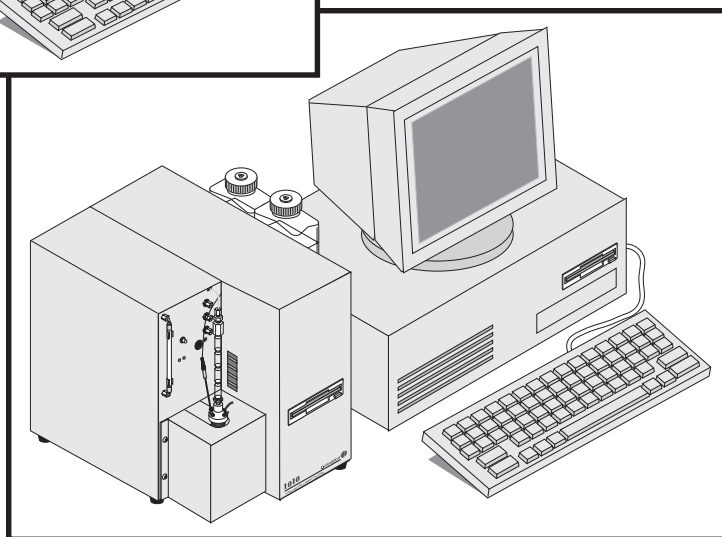
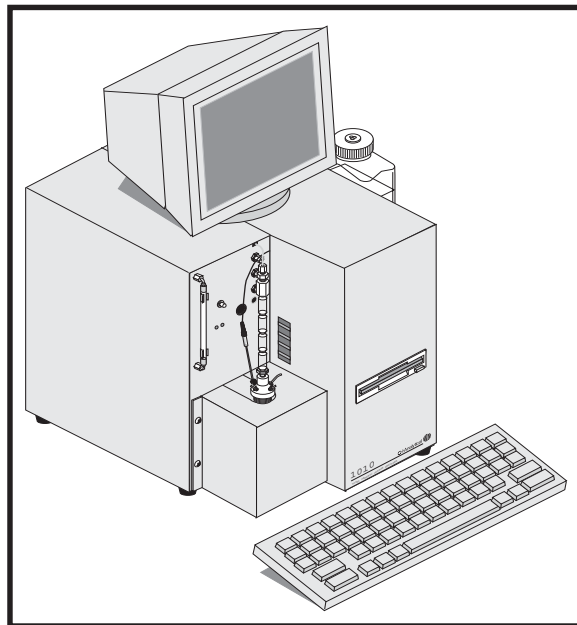




Model 1010 Wet Oxidation Total Organic Carbon Analyzer Operator's Manual



151 Graham Road • P.O. Box 9010 • College Station, Texas 77842-9010
Telephone (979) 690-1711 • FAX (979) 690-0440 • www.oico.com



Notice

The information contained in this document may be revised without notice.

OI Analytical shall not be liable for errors contained herein or for incidental, or consequential, damages in connection with the furnishing, performance, or use of this material.

No part of this document may be reproduced, photocopied, or translated to another language, without the prior written consent of OI Analytical.

Revision 9.4 — April 2003

OI Analytical has been licensed to sell MS-DOS 5.0 under an agreement with Annabooks.

The OI Analytical Total Organic Carbon Analyzer is protected under U.S. Patent #4,619,902.

OI Analytical has applied for patent protection for the Dual Loop Fluid Injector (multi-loop sampling capability).

Centronic is a registered trademark of Centronic Holdings Limited (United Kingdom Co.)

Norprene is a registered trademark of Norton Co.

Windows is a trademark and MS-DOS is a registered trademark of Microsoft Corp.

Swagelok is a registered trademark of Crawford Fittings Co.

Teflon is a registered trademark of E.I. duPont de Nemours & Co., Inc.

Snoop is a registered trademark of Nupro Company.

Drierite is a registered trademark of Hammond Drierite Co.

Kimwipe is a registered trademark of Kimberly-Clark Corp.

OI Analytical Part #273243



Limited Warranty

OI Analytical warrants each Model 1010 Wet Oxidation Total Organic Carbon (TOC) Analyzer and its optional equipment, excluding external personal computers, against defects in materials and workmanship under normal use and service for a period of one (1) year. External personal computers purchased from OI Analytical are warranted through the manufacturer. Equipment installed by OI Analytical is warranted from the installation date; all other equipment is warranted from the ship date. If purchaser schedules or delays installation more than 90 days after delivery, then the warranty period starts on the 91st day from date of shipment. This warranty extends only to the original purchaser. OI Analytical will, at its option, repair or replace equipment that proves to be defective during the warranty period, provided the equipment is returned to OI Analytical at the expense of the purchaser. Parts, labor, and return shipment to the customer shall be at the expense of OI Analytical. Travel costs shall be at the cost of the purchaser.

Software and firmware designed by OI Analytical for use with an external CPU will execute its programming instructions when properly installed on that CPU. OI Analytical does not warrant that the operation of the external CPU, software, or firmware will be uninterrupted or error-free.

Consumables, columns, lamps, and high temperature furnaces are warranted for 30 days (parts only) and are not available for coverage under extended warranties or service contracts.

This warranty shall not apply to defects originating from:

- Improper maintenance or operation by purchaser.
- Purchaser-supplied accessories or consumables.
- Modification or misuse by purchaser.
- Operation outside of the environmental and electrical product specifications.
- Improper or inadequate site preparation.
- Purchaser-induced contamination or leaks.

THE FOREGOING WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO ANY WARRANTY OF MERCHANTABILITY, FITNESS, OR ADEQUACY FOR ANY PARTICULAR PURPOSE OR USE. OI ANALYTICAL SHALL NOT BE LIABLE FOR ANY SPECIAL, INCIDENTAL, OR CONSEQUENTIAL DAMAGES, WHETHER IN CONTRACT, TORT, OR OTHERWISE.

Any service requests or questions should be directed to the Technical Support Department at (800) 336-1911. When calling from international locations, please call (979) 690-1711.



Table of Contents

Chapter 1: Introduction

Principle of Operation	1
Features	1
Specifications	3
Compliance and Safety Information	5
Definitions	8
Summary of Method	9
Interferences	11
Reagents	11
Alternate Standards	12

Chapter 2: Description of Components

Model 1010 - Front View	15
Model 1010 - Back View	17

Chapter 3: Installation

General Information	19
Installation of Basic Unit	20
Installation of External Options	22

Chapter 4: Introduction to Firmware

Firmware Screens	23
Startup Screen	24
RUN SCREEN	25
SEQUENCE SCREEN	27
CONFIGURATION SCREEN	29
CALIBRATION RESULTS SCREEN	34
DIAGNOSTICS SCREEN	35

Chapter 5: Operation

Overview	37
Keyboard and Monitor Operation	40
Starting up the Unit	41
Analyzing Reagent Blanks	41
Calibration	42
Running Samples	49
Sequencing	49
Files	50
Analytical Hints	51

Chapter 6: Maintenance

Schedule for Routine Maintenance	55
Nonscheduled Maintenance - Mechanical	63
Nonscheduled Maintenance - Electronic	68

Chapter 7: Troubleshooting

System Performance Symptoms	69
System Component Symptoms	74
Warning Screens	75



Chapter 8: Replacement Parts	
Replacement Parts	77
Supplies and Options	79
Index	
Index	83



Chapter 1

Introduction

The Model 1010 Wet Oxidation TOC Analyzer is a totally automated system for analyzing aqueous samples for total organic carbon (TOC) and total inorganic carbon (TIC). It uses the USEPA-approved persulfate oxidation method for analysis of samples containing 2 ppb to 12,500 ppm of organic carbon.

All OI Analytical TOC Analyzers are available either as stand-alone systems with keyboard and monitor for direct access to instrument parameters and data or with the Windows®-based WinTOC™ control software, which allows complete control from an IBM-compatible PC. This manual describes the operation of the Model 1010 using the keyboard and monitor.

Principle of Operation

Same-sample determinations of TIC and TOC are performed by wet oxidation. TIC is determined by measuring the carbon dioxide released when a sample is acidified. The carbon dioxide is purged from the solution and detected by a nondispersive infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. This mass is proportional to the mass of TIC in the sample. After the sample has been acidified and purged of TIC, sodium persulfate is added. This oxidant quickly reacts with organic carbon in the sample at 100°C to form carbon dioxide. The carbon dioxide is purged from the solution and detected by the NDIR. The resulting mass of carbon dioxide is proportional to the mass of TOC in the sample.

Features

- Automatically analyzes the aqueous samples for TOC in the range of 2 ppbC to 125 ppmC, and TIC in the range of 2 ppbC to 125 ppmC, without sample pretreatment, prepurging, or dilution. By syringe, the Model 1010 can analyze for TOC and TIC up to 12,500 ppmC.
- A multiloop sampling capability is built into the unit to allow introduction of exact volumes of samples. Samples can be introduced in multiples of the loop size (1 mL, 5 mL, 10 mL, or 25 mL) up to 25 mL.
- Analytical range depends on the volume of sample analyzed. Volume is selected and introduced by syringe injection or sample loop. Volume depends on the general range of concentration.
- Analyzes samples with high levels (near saturation) of dissolved solids, including chloride. TOC in various chemical solutions, reagent acids, and caustics can be quantitatively determined.



- Analyzes samples with suspended solids (up to 100 microns diameter) for TOC, so these samples need not be filtered prior to analysis. The method allows quantitative carbon oxidation in the particulates, so a more accurate TOC value is reported.
- Spikes of known carbon mass may be added to samples for “Method of Standard Additions” verification of TOC recovery in hard-to-handle samples such as acids, caustics, brines, and chemical reagents.
- Sample-wetted parts consist entirely of inert materials to minimize carbon contamination during sample introduction, digestion, and purging. The sample-wetted parts are chemically compatible with essentially all solvents, acids, and bases.
- The single-beam photometric system in the infrared detector minimizes influences due to contamination of the measuring cell and vibration. It features better long-term stability and signal-to-noise ratio over conventional dual-beam analyzers. The single-beam photometric system requires no delicate adjustment of optical balance. Dual-chamber detector effectively minimizes the influence due to interfering or contaminating gas components.
- All electronic and mechanical components are accessible when instrument covers are removed for ease-of-maintenance and service. Power status indicator lamps for valves, heaters, and other components driven by DC voltages are provided for easy troubleshooting.
- The Model 1010 is controlled by a microprocessor that regulates temperatures, controls timing sequences, and performs calculations and continuous system diagnostics.
- Analysis results are directly displayed on a printer connected to the Model 1010 or via WinTOC as ppmC and μC for TIC, TOC, and TC.
- Features singlepoint or multipoint (up to 5 points) calibration.
- Retains analysis conditions in memory, including blank values and the calibration constant, without external power. The operator may select analysis conditions or return to default conditions via disk files.
- Conditions of analysis (times, temperatures, and volumes) may be displayed on the screen.
- Automatically samples from a bottle or flowing stream by sample loop to allow regular sampling of process streams and unattended replicate sampling from sample bottles. The built-in timer may be set to sample and analyze at specified time intervals up to once every 24 hours.
- Has several modes of analysis for a single sample, each of which can be selected by key entry. These include: TIC and TOC; TIC only; TC only; and TOC only.



- A sample ID number, which increases incrementally for each successive sample, may be preset.
- Replicates of blanks, samples, and standards, as well as the number of samples and standards, can be programmed for unattended analysis.
- Sequences of blanks, standards, samples, and check standards can be programmed for unattended calibration.
- TIC, TOC, and TC sample react times and detect times can be extended for difficult sample analyses.
- Low and high concentration TOC, TIC, and TC set points can be entered, and displayed on the screen.

Specifications

Principle Applications

- Standard Method 5310D
- USEPA 415.1
- USEPA 9060
- ASTM D4839
- ASTM D4779
- USP <643>
- Cleaning validation
- Drinking water
- Groundwater
- Wastewater
- Seawater
- Ultrapure water
- Cooling water
- Boiler feedwater
- Pharmaceutical process water
- Semiconductor process water

General Specifications

Dimensions

- Analyzer: 17" H x 13.5" W x 19" D
- With keyboard and monitor: 24.75" H x 13.5" W x 19" D

Weight

- 46 lbs (20 kg)

Alarm Relay Contacts

- 10 A/240 VAC
- 8 A/24 VDC



Performance Specifications

Analysis Modes

- TIC/TOC, TIC, TOC, and TC

Range*

Loop Sampling

- TOC: 2 ppbC–125 ppmC (0.05 µgC–125 µgC)
- TIC: 2 ppbC–125 ppmC (0.05 µgC–125 µgC)

Syringe Injection

- TOC: 100–12,500 ppmC (10–125 µgC)
- TIC: 100–12,500 ppmC (10–125 µgC)

Precision*

- TOC: Greater than $\pm 2\%$ or 2 ppbC
- TIC: Greater than $\pm 2\%$ or 2 ppbC

Time of Analysis

- TOC and TIC: 6–9 min (typical)

Sample Introduction

- Syringe
- Loop sampling
- On-line
- Vial autosampler

Sample Size

- By syringe: 10 µL–1 mL
- By loop: 1–25 mL in 1-mL increments, 5–25 mL in 5-mL increments, 10–20 mL in 10-mL increments, and 25 mL in 25-mL increments

Communications

- Parallel and serial communications (RS-232-C)
- Auxiliary output for optional equipment

Environmental

- Temperature: 10°–40°C
- Relative Humidity: <90%

* The range and precision of analysis are affected by sample introduction, cleanliness of sample containers, purity, gas purity, and operator skill.

Requirements

Gas Requirements

- Nitrogen, 99.98% purity or better, 50–60 psig (345–413 kPa)
- Consumption: <400 mL/min in normal operation



Power Requirements

- 100 ($\pm 10\%$) VAC 50/60 Hz
- 110–125 ($\pm 10\%$) VAC 50/60 Hz
- 210–240 ($\pm 10\%$) VAC 50/60 Hz (optional)

Options

User Interface Options

- Keyboard and monitor (9-inch)
- WinTOC (Windows-based) software for PC control (computer not included)

Other

- Printer
- Vial autosampler
- Halide scrubber
- Solids TOC Analyzer

Printer Specifications

- Dot matrix
- Centronics parallel interface
- 9-pin printhead
- Draft mode: 300 cps
- NLQ mode: 50 cps
- IBM-/Okidata-/Epson-compatible

Compliance and Safety Information

The OI Analytical Model 1010 meets the following electromagnetic compliance certification:

EN50082-1
EN55011 Group 1 Class A

The Model 1010 has been designed and tested in accordance with recognized safety standards and designed for use indoors. Using the instrument in a manner not specified by the manufacturer may impair the instrument's safety protection. Whenever the safety protection of the Model 1010 has been compromised, disconnect the instrument from all power sources and secure the instrument against unintended operation.

The exposure to personal hazards for the Model 1010 and the methodology employed have not been precisely defined. The instructions for installation and operation given in this manual are believed to be a thorough account for proper and safe operation. However, it is the responsibility of each laboratory for maintaining the Model 1010 in a condition suitable for safe use.



Operator Precautions

For operator safety, pay attention to **WARNING** and **CAUTION** statements throughout the manual.

- A **WARNING** indicates a condition or possible situation that could result in physical injury to the operator.
- A **CAUTION** indicates a condition or possible situation that could damage or destroy the product or the operator's work.

Warnings and precautions in this manual or on the instrument must be followed during operation, service, and repair of the instrument. Failure to follow these warnings and precautions violates the safety design standards and intended use of the instrument. OI Analytical will not be liable for the operator's failure to comply with these warnings and precautions.

The Model 1010 must be connected to the AC power supply mains through a three-conductor power cord with the third wire firmly connected to an electrical ground at the power outlet. Any interruption of the grounding conductor or disconnection of the protective earth terminal could cause a shock that could result in personal injury.

General Precautions

- Disconnect the AC power cord before removing any covers.
- Replace or repair faulty or frayed insulation on power cords.
- Perform periodic leak checks on supply lines, fittings, and pneumatic plumbing.
- Arrange gas lines so they can not become kinked, punctured, or otherwise damaged, and will not interfere with foot traffic.
- Turn off the main power switch and disconnect the main power cord before using a liquid solution to locate leaks.
- Do not restrict airflow on the back or left side of the unit.
- Wear safety glasses to prevent possible eye injury.
- Do not replace blown fuses inside the Model 1010. Only trained service personnel should access the interior right bay of the Model 1010.
- Do not perform unauthorized modifications or substitute parts that are not OI Analytical original parts to the instrument. Any unauthorized modifications or substitutions will void the warranty.
- Verify that all heated areas have cooled before handling or wear adequate hand protection to prevent burns.



Chemical Precautions

- The toxicity or potential health risk hazard of chemicals used in this method have not been precisely defined. However, all chemicals and samples used should be treated as a potential health risk, and exposure to the materials should be minimized. Each laboratory is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals and associated equipment used in this method.
- Phosphoric acid has been identified as a corrosive and toxic material. Pure material and diluted solutions of this compound should be handled in a manner consistent with OSHA regulations. Appropriate skin and eye protection should be worn when the operator handles any materials containing this substance.
- The salts of peroxydisulfate ($\text{Na}_2\text{S}_2\text{O}_8$ and others) and solutions containing these salts have been identified as strong oxidizers, and corrosive and toxic materials. Pure materials and diluted solutions of these compounds should be handled in a manner consistent with OSHA regulations. Appropriate skin and eye protection should be worn when the operator handles any materials containing these salts. Caution should be exercised when handling these salts or solutions containing these salts in the presence of organic materials, which could result in accidental contact.
- Potassium biphthalate and sodium carbonate have been identified as chemical irritants to human skin and eyes. Pure materials and stock solutions of these compounds should be handled in a manner consistent with OSHA regulations. Appropriate skin and eye protection should be worn when the operator handles any materials containing these substances. The operator should avoid exposure to fumes or dust.
- Nitrogen has been identified as an asphyxiant. This and its compressed gas cylinder should be handled and stored in a manner consistent with OSHA regulations. Adequate ventilation should be maintained in areas where this material is used and stored. The operator should avoid prolonged exposure to high concentrations of this gas.
- Oxygen has been identified as an oxidizer. This gas and compressed gas cylinder should be handled and stored in a manner consistent with OSHA regulations. Open flames and easily-ignited materials should not be brought in contact with the pure gas except under approved, controlled conditions by the operator. The operator should also avoid prolonged exposure to high concentrations of this gas.

Compressed Gas Cylinders Precautions

- Compressed gases should be stored and handled strictly in accordance with relevant safety codes.
- Fasten all cylinders securely to an immovable structure or permanent wall.



- Store or move cylinders only in a vertical position. Do not move or transport cylinders with regulators attached.
- Use only approved regulators and tubing connections.
- Connect cylinders to instruments with pressure ratings that are significantly greater than the highest outlet pressure from the regulator.

Safety Symbols

The following symbols are located on the instrument:



See accompanying instruction for more information.



Indicates a hot surface.



Indicates hazardous voltages.



Indicates earth (ground) terminal.

Definitions

TC - Total Carbon: TC is all of the carbon in a sample, including inorganic, organic, and volatile carbon, as may be present. TC is reported as total mass of carbon per unit of sample (mgC/L, etc.).

TIC - Total Inorganic Carbon: TIC is all of the carbon in a sample that is converted to carbon dioxide after sample acidification. TIC includes all dissolved carbon dioxide, bicarbonate, and carbonate species, and is reported as total mass of carbon per unit of sample (mgC/L, etc.).

TOC - Total Organic Carbon: TOC is all of the carbon in organic compounds that is converted to carbon dioxide by oxidation, after inorganic carbon has been removed. Although TOC in water samples should ideally include carbon in volatile materials, most laboratories report TOC analyses of samples where volatiles have been previously removed. The widely accepted methods involving persulfate oxidation call for acidification and purging to remove inorganic carbon before oxidation of organics. Purging can also remove volatile organics before oxidation, although the results are still generally accepted as TOC. Volatiles can be included in TOC by separately measuring TC and TIC and calculating TOC by difference.

DOC - Dissolved Organic Carbon: DOC is organic carbon determined by the analysis of aqueous samples that have been filtered through 0.45-micron filters. DOC is reported as total mass of carbon per unit of sample (mgC/L, etc.).



SOC - Suspended Organic Carbon: SOC is organic carbon determined by the analysis of particles captured by the filtration of aqueous samples through a 0.45-micron filter. SOC is sometimes called particulate organic carbon. It is reported as total mass of carbon.

POC - Purgeable Organic Carbon: POC is organic carbon purged from solution by a stream of gas under a specific set of purging conditions. As of this writing, specific conditions have not yet been standardized.

NPOC - Nonpurgeable Organic Carbon: NPOC is organic carbon that remains in solution after a sample has been purged by a gas stream under a specific set of purging conditions. NPOC is often reported as TOC due to popular methods requiring acidification and purging of TIC prior to oxidation of organics. This substitution is valid for samples containing negligible volatile or purgeable organic compounds.

ppmC - parts-per-million Carbon: Parts-per-million carbon are mass units of carbon per million sample mass units ($\mu\text{gC/g}$). In aqueous samples this is generally the same as mgC/L .

ppbC - parts-per-billion Carbon: Parts-per-billion carbon are mass units of carbon per billion sample mass units (ngC/g). In aqueous samples this is generally the same as $\mu\text{gC/L}$.

Reagent Blank: The reagent blank is the detector response in area counts generated from an analysis sequence (with reagents) without introduction of a sample or standard. The response is due to carbon contamination in the reagents, gas, digestion vessel, and/or tubing.

Standard: A standard is any sample with a known amount of added carbon.

Water Blank: The water blank is the response of the analyzer to the carbon content of water when it is analyzed. This value will be taken into account by analyzing reagent water as a zero concentration standard during calibration.

Summary of Method

Total Inorganic Carbon (TIC) is determined by measuring the carbon dioxide released by sample acidification. As pH of the sample is lowered, carbonate and bicarbonate ions are converted to dissolved carbon dioxide. This carbon dioxide is purged from solution and carried into a nondispersive infrared detector (NDIR) calibrated to directly display the mass of carbon dioxide detected. This mass is equivalent to the mass of TIC in the sample. The concentration of TIC is calculated by dividing this mass by the sample volume.

Total Organic Carbon (TOC) is determined by measuring the carbon dioxide released by chemical oxidation of the organic carbon in the sample. After the sample has been acidified and purged of TIC, sodium persulfate ($\text{Na}_2\text{S}_2\text{O}_8$), a strong oxidizer, is added. This oxidant quickly reacts with organic carbon in the sample at 100°C to form carbon dioxide. When the oxidation reaction is complete,



the carbon dioxide is purged from the solution and detected as described for TIC. The resulting carbon mass in the form of carbon dioxide is equivalent to the mass of organic carbon originally in the sample.

Total Carbon (TC) is determined by measuring the carbon dioxide released by complete oxidation of all carbon present in the sample (inorganic and organic). For this analysis, first add acid and persulfate to the sample and allow a specific reaction time to convert all carbon present to carbon dioxide. When the reaction is complete, the resulting carbon dioxide is purged from the solution and detected as described for TIC. The resulting carbon mass detected in this analysis is equivalent to one of two sums. Either the sum of TIC + POC + NPOC, or if the sample was preacidified and sparged before analysis, the carbon mass, will be due only to the dissolved organic carbon (e.g. NPOC).


Nondispersive Infrared Detector (NDIR): The infrared gas analyzer measures gas concentration based on the principle that each type of gas component shows a unique absorption spectrum in the infrared region. The IR detector contains an infrared light source, a beam chopper, a measuring cell, and a detector filled with a gas mixture containing the gas component to be measured (CO_2).

The light source emits infrared light in all directions. The infrared light emitted backward is reflected and added to the infrared light emitted forward. The infrared light beam formed passes through the measuring cell and is partially absorbed, or attenuated, by any CO_2 present as a sample passes through. The beam then reaches the front chamber of the detector. Both the front and back detector chambers are filled with a gas mixture containing the gas component to be measured (CO_2). The infrared light beam is partially absorbed in the front chamber and residual light is absorbed in the back chamber, increasing pressure in both chambers. The front chamber pressure increases more than the back chamber pressure because of a greater amount of radiation entering the front chamber (it attenuates radiation to the back chamber); slight gas flow is produced through a path connecting the two chambers.

When the measuring cell contains an interfering gas component showing an infrared absorption spectrum overlapped with that of CO_2 , the interfering gas component also causes pressure increases in the front and back detector chambers. In this case, the pressure increases are identical because the front chamber contains no interfering gas component to attenuate radiation. Thus, no gas flow between cells is produced when interference is introduced to the cell.

Between the infrared light source and the measuring cell is a chopper blade that rotates to interrupt the infrared light beam at a regular frequency (10 Hz), so that it reaches the detector chamber intermittently. Pressure then rises periodically in the chambers to produce a slight flow pulsation. The amplitude at the flow pulsation is greatest when no CO_2 is flowing through the measuring cell. The flow pulsation is converted into AC electrical signals by a microflow sensor located in the path connecting the chambers. The AC signals are amplified and rectified into DC voltage signals to be supplied to the microprocessor. The voltage output is continuously linearized with respect to the mass of carbon (volume of CO_2) momentarily flowing through the cell.




WARNING:
The toxicity or potential health risk hazard of chemicals used in this method have not been precisely defined. All chemicals and samples used should be treated as a potential health risk, and exposure to the materials should be minimized. Each laboratory is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals and associated equipment used in this method.

Interferences

Method Interferences

Carbon is ubiquitous in nature, so reagents, water, and glassware cannot be cleaned completely of it. Method interferences (positive bias) may be caused by contaminants in the gas, dilution water, reagents, glassware, or other sample processing hardware such as homogenizers. All of these materials must be routinely demonstrated to be interference-free under the analysis conditions by running reagent blanks. Use high purity or purified reagents and gases to help minimize interference problems.

Calibration Interferences

With most TOC instruments, a correction for TOC in the dilution water used for calibration standards must be considered, especially for standards below 10 ppmC. The Model 1010 is designed to eliminate this problem. Various instrument calibration techniques will be discussed in Chapter 4, "Operation."

Interference By Non-CO₂ Gases: The infrared detector is sensitized to CO₂ and accomplishes virtually complete rejection of response from other gases which absorb energy in the infrared region.

Interferences in Sampling: For most accurate analyses, sampling containers should be free of organic contaminants. Plastic bottles can bleed carbon into water samples, especially when they are new, or when they are used for low-level samples (less than 200 ppbC). Any new bottles (especially plastic) should ideally be filled with clean water for a period of several days or boiled in water for a few hours before use. Pyrex bottles should be washed and muffled at 400°C before first use. Sample TIC and POC can be affected by exposure to the atmosphere, as well as sample TOC below about 50 ppbC. In these cases, sampling bottles should be kept closed when possible, and autosampler vials should be equipped with septa for needle-piercing by the autosampler.

Reagents

Reagent Water: Distilled or deionized water containing TOC of less than 200 ppbC is recommended.

Sodium Persulfate: The optimum concentration of sodium persulfate solution depends on the range of carbon to be detected, as shown in the table below.

Carbon Range	Na ₂ S ₂ O ₈ Concentration
0–125 µgC	100 g/L*

*Use 200 g/L when analyzing difficult matrices (e.g., sea water brines, etc)



Note: It is also possible to change the volume of reagent used per analysis to operate within these ranges.

Prepare a 100 g/L or 200 g/L solution of sodium persulfate by dissolving 100 or 200 g $\text{Na}_2\text{S}_2\text{O}_8$ into reagent water (1 L total volume). Stirring may be necessary, but do not heat. Transfer a portion of this solution to the appropriate reagent bottle provided with the Model 1010. Shelf life is approximately three weeks. Sodium persulfate and reagent are available from OI Analytical.

Phosphoric Acid (5% vol/vol): Prepare a 5% by volume solution of phosphoric acid by adding 59 mL of ACS Reagent Grade 85% H_3PO_4 to reagent water (1 L total volume).

The acid solution may be purified, if high organic contamination of the solution is suspected, by adding 10 mL of the persulfate solution and immersing the vented container in boiling water for at least two hours. The persulfate will oxidize any TOC in the solution and then completely autodegrade in two hours at 100°C. The cooled solution should then be purged for several minutes to remove any CO_2 from oxidation of organics. The decrease in reagent blank resulting from this procedure is not generally worth the purification effort unless the acid solution is found to be abnormally high in TOC. Phosphoric acid and reagent are available from OI Analytical.

Potassium Biphthalate Stock Solution (KHP) (1000 ppmC): Prepare a stock solution by adding 2.128 g of KHP (previously dried to constant mass at 110°C) into a 1000 mL volumetric flask. Dilute to volume with reagent water. Lower concentration standards can be prepared from this stock. It contains 1.0 $\mu\text{gC}/\mu\text{L}$. Shelf life is approximately three weeks. KHP and stock solution are available from OI Analytical.

Sodium Carbonate Stock Solution (Na_2CO_3) (1000 ppmC): Prepare a stock solution by adding 8.826 g of Na_2CO_3 (previously dried to constant mass at 110°C) to a 1000-mL volumetric flask. Dilute to volume with reagent water. Lower concentration standards can be prepared from this stock. It contains 1.0 $\mu\text{gC}/\mu\text{L}$. Shelf life is approximately three weeks. Na_2CO_3 and stock solution are available from OI Analytical.

Alternate Standards

Other standards can be used to calibrate the Model 1010. It is recommended that if standards other than potassium biphthalate or sodium carbonate are used, that these other standards should be easily oxidized.

United States Pharmacopeia (USP) Standards

Below are standards that are proposed for use during calibration of the TOC. This information does not replace or amend information in a formal method.

Sucrose ($\text{C}_{12}\text{H}_{22}\text{O}_{11}$) (1,000 ppmC TOC): Prepare a stock solution by adding 2.375 g of $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ (previously dried to constant mass at 110°C) into a



1,000-mL volumetric flask. Dilute to volume with reagent water. Lower concentration standards can be prepared from this stock, which contains 1.0 $\mu\text{gC}/\mu\text{L}$.

Sucrose ($\text{C}_{12}\text{H}_{22}\text{O}_{11}$) (10 ppmC TOC): Prepare a stock solution by adding 47.5 mg of $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ (previously dried to constant mass at 110°C) into a 2,000-mL volumetric flask. Dilute to volume with reagent water. Lower concentration standards can be prepared from this stock.

Sucrose ($\text{C}_{12}\text{H}_{22}\text{O}_{11}$) (500 ppbC TOC): Prepare a stock solution by adding 1.19 mg of $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ (previously dried to constant mass at 105°C) into a 1,000-mL volumetric flask. Dilute to volume with reagent water. Lower concentration standards can be prepared from this stock.

1,4-benzoquinone (parabenzoquinone) ($\text{C}_6\text{H}_4\text{O}_2$) (10 ppmC TOC): Prepare a stock solution by adding 30.0 mg of $\text{C}_6\text{H}_4\text{O}_2$ into a 2,000-mL volumetric flask. Dilute to volume with reagent water. Lower concentration standards can be prepared from this stock.

1,4-benzoquinone (parabenzoquinone) ($\text{C}_6\text{H}_4\text{O}_2$) (500 ppbC TOC): Prepare a stock solution by adding 0.75 mg of $\text{C}_6\text{H}_4\text{O}_2$ into a 1,000-mL volumetric flask. Dilute to volume with reagent water. Lower concentration standards can be prepared from this stock.



Notes



Chapter 2

Description of Components

Model 1010 - Front View

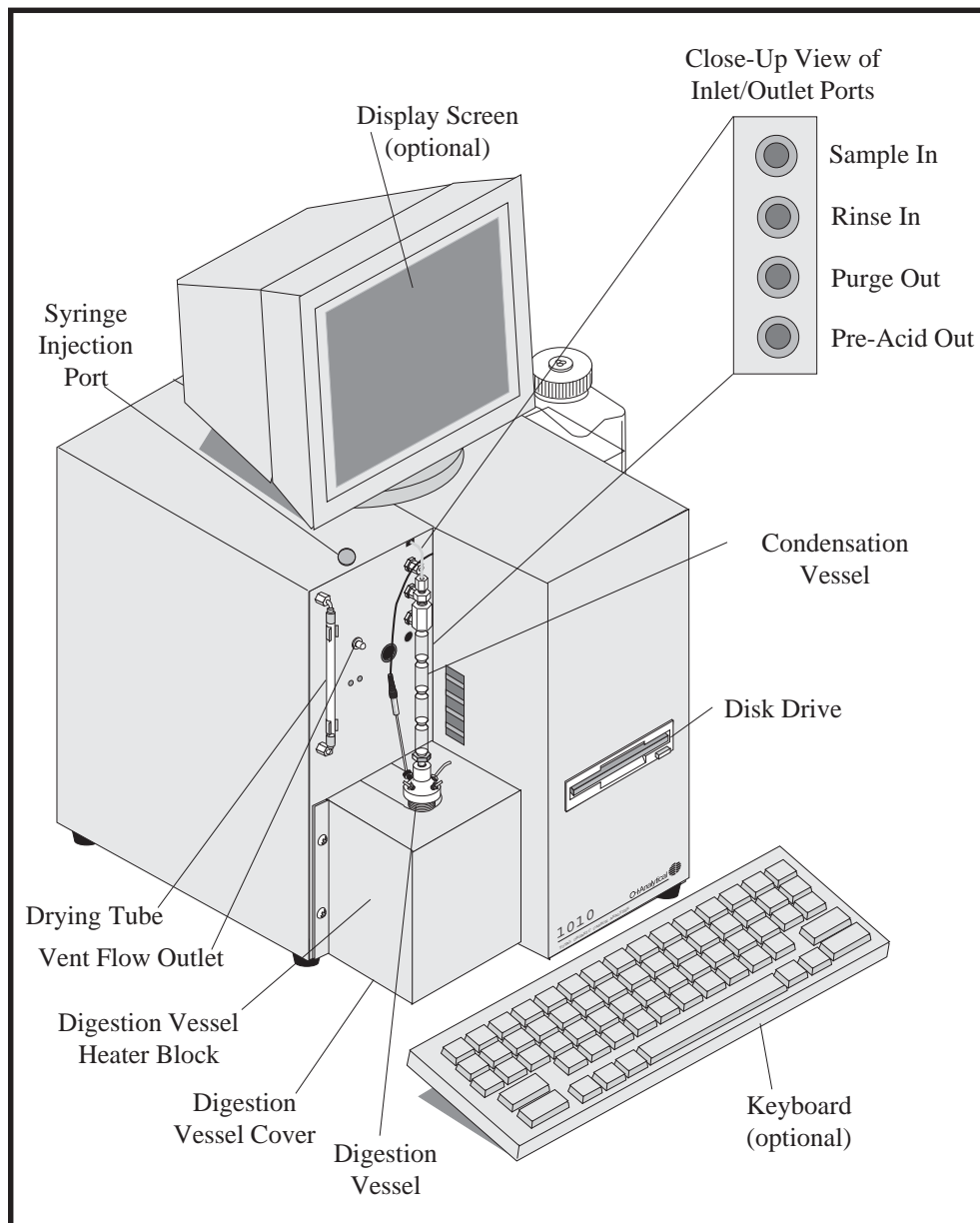


Figure 2.1. Model 1010 - Front View

Condensation Vessel allows for condensation of water vapor from the sample.

Digestion Vessel is a quartz vessel into which reagents are added to the sample for TIC/TOC analysis.



Digestion Vessel Cover is the protective cover over the digestion vessel, its heating elements, and electrical components.

Digestion Vessel Heater Block is for heating the digestion vessel to promote oxidation.

Disk Drive is a standard 3.5-inch computer disk drive. The operating system for the TOC analyzer is loaded and upgraded from this disk drive. The program disk must be removed from the disk drive when the Model 1010 is operating.

Display Screen is a 9-inch monochrome monitor used to view selected settings and parameters. (Not needed if WinTOC is used.)

Drying Tube indicates when moisture is present by changing color (from blue to pink).

Keyboard is used to enter settings and select parameters displayed on the display screen. (Not needed if WinTOC is used.)

Pre-Acid Outlet (Pre-Acid Out) is the outlet port for acid when the preacidification module is used.

Purge Line Outlet (Purge Out) is the line for nitrogen gas to purge samples.

Rinse Line Inlet (Rinse In) is the inlet line for rinse water to rinse the system between sample or replicate analyses.

Sample Inlet (Sample In) is the inlet port for sample introduction by sample loop. The sample may be aspirated into the sample loop through this line from a sample vessel or an autosampler, or may be sampled intermittently from a flowing stream.

Syringe Injection Port is used for introduction of the sample into the Model 1010 via syringe.

Vent Flow Outlet is the gas outlet of the system.



Model 1010 - Back View

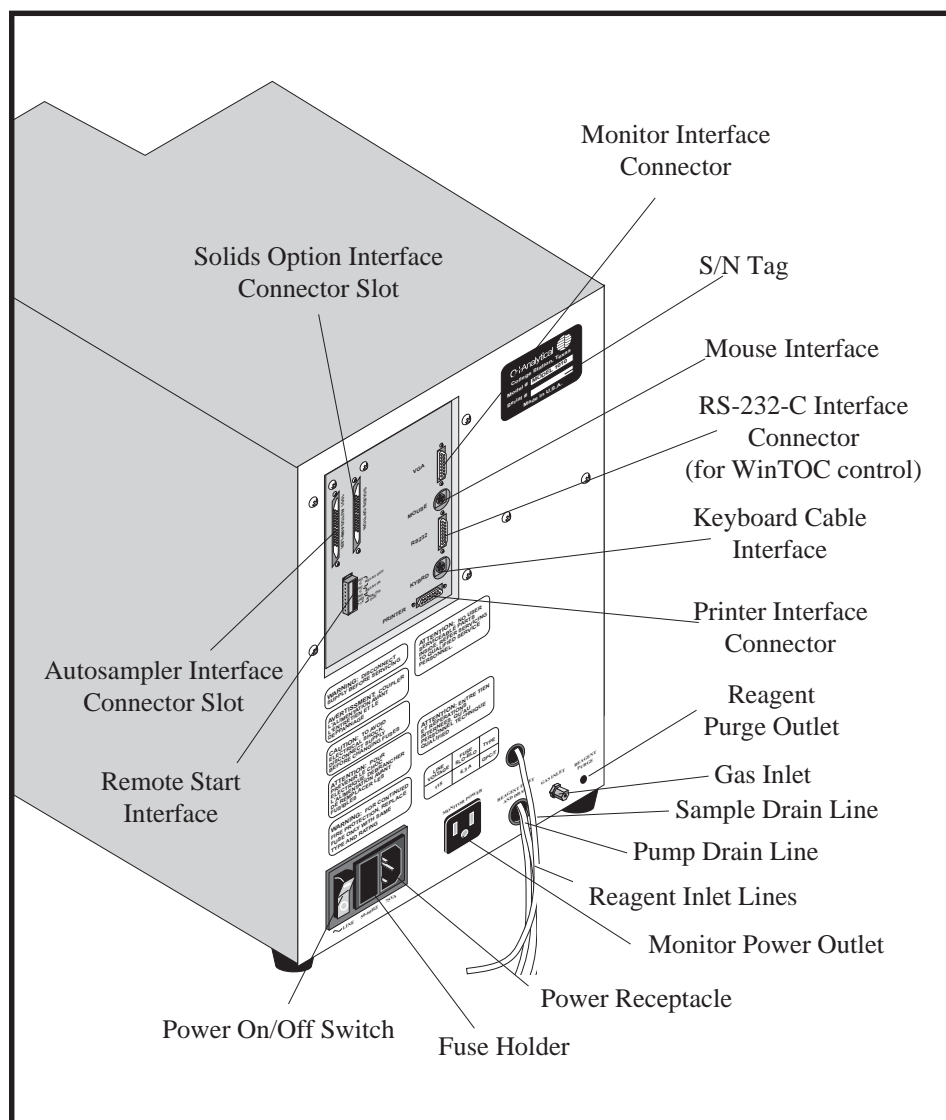


Figure 2.2. Model 1010 - Back View

Autosampler Interface Connector Slot allows interface to an OI Analytical autosampler via an Autosampler Interface Connector. This connector interfaces to a standard 25-pin male connector. If the Model 1010 does not have an autosampler, this slot is empty.

Card Cage Assembly houses printed circuit boards that allow interface to external devices.

Fuse Holder houses the main fuse which protects the Model 1010 from short circuiting.

Gas Inlet provides an inlet for nitrogen gas.



WARNING:
Do not attempt to connect any other electrical device to the Monitor Power Outlet.

Information and Warning Labels warn the operator of potential hazards associated with improper use of the Model 1010 and inform the operator of voltage requirements.

Keyboard Cable Interface allows connection to the Model 1010 keyboard.

Monitor Interface Connector allows interface to the monitor used with the Model 1010.

Monitor Power Outlet allows output power for the Model 1010 monitor (only).

Power On/Off Switch is the power control switch. A power-up self test occurs when turning on the Model 1010.

Power Receptacle connects the Model 1010 to an appropriate power source via a cable provided in the start-up kit.

Printer Interface Connector is a parallel interface to an external printer. This connector interfaces to a standard computer printer interface cable with a 25-pin male connector on one end and a Centronics parallel printer connector on the other.

Pump Drain Line provides an exit for sample that has overflowed the sample loop. Route this line to an appropriate waste receptacle.

Reagent Inlet Lines are the inlet lines for the reagents (oxidant and acid) from the reagent bottles to the Model 1010.

Reagent Purge Outlet provides gas to purge the reagents in the reagent bottles.

RS-232-C Interface Connector allows interface to an IBM-compatible personal computer, when using WinTOC. This connector interfaces with a standard 25-pin male connector.

Sample Drain Line allows waste from the analysis to exit the Model 1010. Route this drain line to an appropriate waste receptacle.

S/N Tag displays the model number and serial number of the Model 1010.



Chapter 3

Installation

In Chapter 2, “Description of Components,” the names and functions of the various Model 1010 Wet Oxidation TOC Analyzer components were outlined. These names will refer to components involved in the installation of the Model 1010.

This chapter includes step-by-step procedures for properly installing the Model 1010 and its optional equipment. The chapter begins with general information, then discusses materials needed for installation that are not included with the basic instrument. The operator should gather the materials outlined here before attempting the installation, then proceed step-by-step through the instructions, beginning with “Installation of Basic Unit.”

General Information

After opening the shipping container, unpack the instrument and check the items against the component list. If any damage is apparent, notify the carrier immediately. Save all packing materials until proper operation of the detector has been verified.

Note: All instruments that are returned to OI Analytical for service or warranty repair must be shipped in the instrument’s original OI Analytical box with its packing materials. *If instruments are damaged due to improper shipping, OI Analytical will not be responsible for the cost of repairs.* If there is no access to proper shipping materials, contact OI Analytical Order Entry Department at (800) 336-1911 or (979) 690-1711.

Internal vs. External Options

The Model 1010 is shipped with any external options packed in separate boxes and any internal options already installed within the Model 1010.

Removing Instrument Covers

The right bay contains all electronic components except the NDIR and the left bay contains all components that contact the liquid sample. These components may be exposed by removing the left or right bay covers. Covers are removed in the following manner:

1. Turn off the power and disconnect the Model 1010 from its power source.
2. Remove the monitor from the top of the Model 1010.



3. Remove the reagent bottles from the tray on the right side of the Model 1010.
4. Remove the screws located on or near the top of the left and right covers of the Model 1010.
5. Slide the instrument covers toward the back of the Model 1010 and lift them upwards.

Installation of Basic Unit

Materials Needed Before Installation

For a typical installation, the operator must have several items on hand before installing the Model 1010. The following are required materials for installation:

- Source of high purity reagent water, TOC 200 ppbC or less.
- For low-level analysis, reagent water must be less than 50 ppbC.
- 5% phosphoric acid reagent.
- 100 g/L or 200 g/L sodium persulfate reagent.
- Potassium biphthalate (TOC standard), primary standard crystals, and/or 1,000 ppmC standard solution or suitable TOC standard.
- Nitrogen (99.98% purity or better).
- Appropriate wrenches for gas connections.
- 1/8" O.D. x 0.063" I.D. gas tubing. Twenty feet of Teflon® tubing is suggested.

Optional Materials

- Double-stage nitrogen regulator (0–60 psig). (May not be necessary if a nitrogen generator is used.)
- One 1/4" MNPT x 1/8" Swagelok® adapter for adapting most gas regulators to 1/8" O.D. gas tubing. (May not be necessary if a nitrogen generator is used.)
- Teflon pipe tape (for pipe threads only). (May not be necessary if a nitrogen generator is used.)



Gas and Fluid Connections

Note: The basic Model 1010 has a start-up kit. Some options also contain start-up kits.

1. After opening the instrument crate, remove the start-up kit(s) and inspect for completeness. Listings of components are included in the kit(s).
2. Remove the Model 1010 from the crate and locate it near a suitable gas source and electrical power. The power cord is 72 inches (180 cm).

Note: If the bottle tray rack is already in place, skip step 3.

3. Install the reagent bottle tray (open side up) on the right side of the Model 1010 using the two screws found in the mounting holes on the right side of the Model 1010.
4. Connect the one gas line (nitrogen) to the gas inlet. See Chapter 2, “Description of Components” for a view of the Model 1010 back panel and a description of components.
5. Connect the other end of the gas line to an appropriate nitrogen gas source.
6. Remove the plugs from the vent, sample in, purge out, and rinse ports on the front panel of the Model 1010. (See Chapter 2, “Description of Components.”)

Note: If the plugs are not removed, the Model 1010 will not function properly.

7. Route the drain tubes to a waste bottle or other receptacle.
8. Turn on the nitrogen gas flow and immediately adjust the regulator pressure to 50–60 psi. Check for supply gas leaks with Snoop[®] or other suitable leak detector.
9. Fill the reagent bottles with reagents prepared according to instructions (see Chapter 1, “Introduction”).
10. Screw the acid reagent bottle cap marked H_3PO_4 (phosphoric acid) onto the acid bottle and the persulfate reagent bottle cap marked $\text{Na}_2\text{S}_2\text{O}_8$ (sodium persulfate) onto the persulfate bottle. (The $\text{Na}_2\text{S}_2\text{O}_8$ inlet line should have a filter installed on the end.)
11. Confirm that the solutions in the reagent bottles are being purged.
12. Place the reagent bottles into the bottle tray rack.
13. Connect the power cord to the corresponding receptacle on the back of the Model 1010 and plug it into an appropriate voltage source.
14. Install any external options before initial power-up.



Installation of External Options ████████████████████

Printer (optional when using WinTOC)

1. Follow the instructions in the printer manual for proper installation of paper, paper guide, and cable.
2. Plug the printer interface cable into the connector labeled “Printer” on the back of the Model 1010.
3. Plug the printer power cord into an appropriate power outlet.
4. Turn the printer power on.

Autosampler

- Follow the instructions in the autosampler manual.

Keyboard and Monitor

1. Remove the keyboard and monitor from packing.
2. Plug the keyboard into the back of the Model 1010, into the connector labeled “Keyboard.” The keyboard connector has a label that reads “TOP” or “UP.” This label should be oriented toward the left side (back view) of the unit (reagent bottle side).
3. Place the keyboard in front of the Model 1010.
4. Place the monitor on top of or beside the Model 1010.
5. Plug the monitor video cable into the back of the Model 1010, into the connector labeled “Video.”
6. Connect the power cord into the corresponding receptacle on the back of the Model 1010.
7. Turn on the monitor power.



Chapter 4

Introduction to Firmware

This chapter provides descriptions of the menus, screens, and commands used to control the Model 1010 TOC Analyzer by keyboard and monitor using the DOS-based control software provided on the Program Disk (“Firmware”). To operate the Model 1010 TOC Analyzer, refer to Chapter 5, “Operation.”

Firmware Screens

The control program consists of nine menus that are accessed by using the function keys on the keyboard. These menus can be divided into three categories: operation, configuration, and diagnostics (see Table 4.1). Each of the five screens are presented in detail in this chapter.

Table 4.1. Menus and Keystrokes for Firmware

Category	Key	Function/Screen
Operation	[F1]	Start/resume the run
	[F2] (pressed once)	Pause the run or sequence
	[F2] (pressed twice)	Abort the run or sequence
	[F3]	RUN SCREEN
	[F10]	Spiral the autosampler tray
Configuration	[F4]	SEQUENCE SCREEN
	[F5]	CONFIGURATION SCREEN
	[F6]	CALIBRATION SCREEN
Diagnostics	[F7]	DIAGNOSTICS SCREEN
	[F8]	Display error messages



Startup Screen

When the TOC Analyzer is initially powered up, the **Startup Screen** appears.

RUN STATUS		TOC 1010 VersionX.XXX				UNIT STATUS			
Stopped						System Ready			
<p>OI ANALYTICAL Total Organic Carbon Analyzer 1010 VersionX.XXX</p>									
F1	F2	F3	F4	F5	F6	F7	F8	F9	F10
START	HOLD	RUN SCRN	SEQ SCRN	CONFIG SCRN	CALIB SCRN	DIAG SCRN			

At the top of this screen, the **RUN STATUS**, **UNIT STATUS**, and the operating software version number are displayed.

RUN STATUS

Informs the operator of the status of the run. The possibilities are **Stopped**, **Running**, **Holding**, **Manual Drain**, and **Waiting For Start**.

UNIT STATUS

Informs the operator if the TOC Analyzer is ready and may include errors or warnings if a problem exists with the instrument. The possibilities are **System Ready**, **Printer Error**, **Low Gas Pressure**, and others, which are discussed in Chapter 7, "Troubleshooting."

If any errors or warnings do occur, an error screen is available by pressing [F8]. The error screen will list any errors or warnings until they are remedied. To exit the error screen, press the [Esc] key. When errors or warnings no longer exist, the error screen option will disappear from the bottom of the screen.

The list of function keys and their corresponding functions is displayed across the bottom of the screen. Pressing any of the function keys [F3] through [F7] will remove the Startup Screen and display the selected screen.



RUN SCREEN

When [F3] is pressed, the RUN SCREEN (RUN SCRNR) appears.

RUN STATUS Running		TOC 1010 Version X.XX		UNIT STATUS System Ready					
	Time	Remaining							
TOTAL RUN	00:00	00:00							
STANDBY	00:05	00:05							
STATUS		PARAMETERS							
Signal	0	Sample Size	10 ml						
Sample Temp	000 C	Loop Size	1 ml						
Sample ID	000	Spl Intro	Sipper						
		Spl Mode	TIC/TOC						
SEQUENCE		Std Mode	TOC						
*Sample		Acid Vol	0200 ul						
		Oxidant Vol	1000 ul						
		Rinse	OFF						
Attenuation: 1									
F1	F2	F3	F4	F5	F6	F7	F8	F9	F10
START	HOLD	RUN SCRNR	SEQ SCRNR	CONFIG SCRNR	CALIB SCRNR	DIAG SCRNR			

The RUN SCREEN displays the run parameters, along with a graphical and numerical output from the NDIR detector. The operator can use the [T] key to toggle between the graphical and numerical displays. The only parameter that can be changed on this screen is **Attenuation**. The states that can occur during a typical run are shown in Table 4.2.

Attenuation

The Up [↑] arrow key increases the attenuation of the peak scale and the Down [↓] arrow key decreases the attenuation of the peak scale. The entire chart will be adjusted by the attenuation.

TOTAL RUN Time

Displays the amount of time required to perform the analysis.

TOTAL RUN Remaining Displays the amount of time remaining in the analysis.

[Current State] Time

Displays the total time of the current state.

[Current State] Remaining

Displays the remaining time of the current state.

STATUS

Provides information on the current status of the TOC Analyzer.

Signal

Displays the current signal output of the NDIR detector.

Sample Temp

Displays the actual temperature of the sample in the digestion vessel.

Sample ID

Displays the current sample number or the number of the vial in the Model 1051 Autosampler being analyzed.



Table 4.2. Model 1010 States

State	Description
STANDBY	Idle state of the TOC Analyzer.
SAMPLE INTRO	Instrument is sampling.
TC DETECT	TC is converted to carbon dioxide by combustion, and the carbon dioxide from TC is detected.
TC REACT	Acid and persulfate reagent are added to the sample to convert TIC and TOC to carbon dioxide.
TIC REACT	Acid reagent is added to the sample.
TIC DETECT	TIC is converted to carbon dioxide, and the carbon dioxide from TIC is purged from the sample and detected.
TOC REACT	Persulfate reagent is added to the sample to convert TOC to carbon dioxide.
TOC DETECT	Carbon dioxide from TOC is purged from the sample and detected.
RINSING	Instrument is rinsing.
DRAIN	Sample and reagents are drained from the reaction vessel.

SEQUENCE

Appears if a sequence is running. Lists the analyses to be performed with an asterisk (*) by the current analysis.

PARAMETERS

Informs the operator of certain operational parameters for analysis based on the configuration specified.

Sample Size

Displays the size of the sample being used.

Note: Sample size is not the exact volume of the sample since loop volumes are calibrated on the Model 1010 and variances in loop volumes, tubing lengths, and valve internal volumes will affect the actual loop volume. The true loop volumes are stored in memory so that when a sample volume is entered, the loop volume is multiplied by this volume to establish true sample volume. The true sample volume will be on the printout when the results of the analysis are printed. The true sample volume is used for all calculations.



Loop Size

Displays the current size of the sample loops installed.

Note: This is not the exact volume of the loop since loop volumes are calibrated on the Model 1010 and variances in loop volumes, tubing lengths, and valve internal volumes will affect the actual loop volume. The true loop volumes are stored in memory and are used in calculations for analysis results. The true loop volumes are displayed on the **DIAGNOSTICS SCREEN** and are also found in the header on the printout.

Spl Intro

Informs the operator of how the sample is introduced into the TOC Analyzer: Sipper or Autosampler.

Spl Mode

Displays the mode of analysis to be performed for samples: TIC/TOC, TIC, TOC, or TC.

Std Mode

Displays the mode of analysis to be performed for standards: TC, TIC, or TOC.

Acid Vol/Oxidant Vol

Displays the volume of the reagents used.

Rinse/Sample

Informs the operator if rinsing per sample is ON or OFF.

During analysis, the analysis type being performed appears below the operating software version number. The number of runs and replicates is also listed.

SEQUENCE SCREEN

When [F4] is pressed, the SEQUENCE SCREEN (SEQ SCR) appears.

RUN STATUS Stopped		TOC 1010 Version X.XX				UNIT STATUS System Ready																																									
[L] LOAD [S] SAVE [D] DELETE		SEQUENCE																																													
RUN TYPE [1] Reagent Blank [C] Clear Table [2] Standard [E] Edit Table [3] Sample [M] Modify STD [4] Check Standard		SEQUENCE TABLE																																													
SPL MODE TIC/TOC STD MODE TOC		<table border="1"> <thead> <tr> <th>#</th> <th>Type</th> <th>Qty</th> <th>Reps</th> <th>Start Pos</th> <th>End Pos</th> </tr> </thead> <tbody> <tr> <td>STD#1</td> <td>000.00000 ppm</td> <td>010 ml</td> <td></td> <td></td> <td></td> </tr> <tr> <td>STD#2</td> <td>000.50000 ppm</td> <td>010 ml</td> <td></td> <td></td> <td></td> </tr> <tr> <td>STD#3</td> <td>001.00000 ppm</td> <td>010 ml</td> <td></td> <td></td> <td></td> </tr> <tr> <td>STD#4</td> <td>005.00000 ppm</td> <td>010 ml</td> <td></td> <td></td> <td></td> </tr> <tr> <td>STD#5</td> <td>010.00000 ppm</td> <td>010 ml</td> <td></td> <td></td> <td></td> </tr> </tbody> </table>										#	Type	Qty	Reps	Start Pos	End Pos	STD#1	000.00000 ppm	010 ml				STD#2	000.50000 ppm	010 ml				STD#3	001.00000 ppm	010 ml				STD#4	005.00000 ppm	010 ml				STD#5	010.00000 ppm	010 ml			
#	Type	Qty	Reps	Start Pos	End Pos																																										
STD#1	000.00000 ppm	010 ml																																													
STD#2	000.50000 ppm	010 ml																																													
STD#3	001.00000 ppm	010 ml																																													
STD#4	005.00000 ppm	010 ml																																													
STD#5	010.00000 ppm	010 ml																																													
F1	F2	F3	F4	F5	F6	F7	F8	F9	F10																																						
START	HOLD	RUN SCRN	SEQ SCRN	CONFIG SCRN	CALIB SCRN	DIAG SCRN																																									



The SEQUENCE SCREEN provides access to sequences for building, modifying, or loading. The default tables are empty and can be built by using the options under **RUN TYPE**. Numerical values must be followed by the [Enter] key.

LOAD, SAVE, DELETE Allows the operator to recall sequences by pressing [L] and choosing the title of the sequence to recall, to display a screen listing by pressing [S], and to delete a sequence by pressing [D].

Note: Loading, saving, and deleting files cannot be performed during analysis.

RUN TYPE Allows the operator to select the type of analysis to be performed (**Reagent Blank, Standard, Sample, or Check Standard**). Once the run type has been selected, the quantity of runs and replicates can be entered. The start and end positions are always "1" for Sipper Mode and can be specified for Autosampler Mode.

Clear Table Allows the operator to clear the sequence table immediately and completely by pressing [C].

Edit Table Allows the operator to edit the sequence table by pressing [E]. The operator can move to the field to be edited using the arrow keys and complete editing by pressing [Esc].

Modify STD Allows the operator to modify the standards or the standard type by pressing [M]. The operator can move to the field to be edited using the arrow keys and complete modifying by pressing [Esc].

STANDARDS Contains the type (TC, TIC, or TOC) and the list of standards 1–5. Concentration and volume can be entered for each standard. These can be programmed into memory to be recalled when standards are analyzed.

SEQUENCE TABLE Allows the operator to program the TOC Analyzer to perform multiple types of analyses. Once a sequence has been programmed, it can be saved.

Qty Displays the number of samples to run with the specified number of replicates. This value is related to **Start Pos** and **End Pos** when using an autosampler. The range is 1 to 999 and is limited by the number of vials when an autosampler is used.

$$\text{Qty} = \text{End Pos} - (\text{Start Pos} + 1)$$

Qty is forced to 1 for standards and check standards.



Reps

Displays the number of replicates to run for each sample. The range is 1–15 for samples and 1–10 for standards and check standards.

Start Pos

Displays the starting vial number for the current sample set. The range is 1 to **End Pos**. This field is not used for Sipper Mode.

End Pos

Displays the ending vial number for the current sample set. The range is **Start Pos** to the autosampler tray maximum (53 or 88).

CONFIGURATION SCREEN

When [F5] is pressed, the CONFIGURATION SCREEN (CONFIG SCR�N) appears.

RUN STATUS Stopped		TOC 1010 Version X.XXX		UNIT STATUS System Ready					
CONFIGURATION									
Sample ID	000	[L] LOAD [S] SAVE [D] DELETE	PRINTER						
Sample Size	5.000 ml		Printer Enable	ON					
Loop Size	1 ml		Print Method	ON					
REAGENT VOLUMES			ALARMS						
Acid	0200 ul	SPL MODE	TIC/TOC	Enabled	OFF				
Oxidant	1000 ul	(Total Time)	00:00	TIC Low	0.000				
TIME (total)		STD MODE	TOC	TIC High	0.000				
TIC React	00:05	(Total Time)	00:00	TOC Low	0.000				
TIC Detect	00:05	SAMPLE INTRODUCTION		TOC High	0.000				
TOC React	00:05	Sipper		TC Low	0.000				
TOC Detect	00:05			TC High	0.000				
TC React				CARBON MASS ALARMS					
TC Detect				Enabled	OFF				
Auto Repeat	00:00:00			CALIBRATION RESULTS					
RINSE				Allow editing?	OFF				
Rinse Volume	15 ml			Date	04 / 13 / 1995				
Per Rep	0			Time	12:55				
Per Sample	0								
F1	F2	F3	F4	F5	F6	F7	F8	F9	F10
START	HOLD	RUN SCR�N	SEQ SCR�N	CONFIG SCR�N	CALIB SCR�N	DIAG SCR�N			

The CONFIGURATION SCREEN provides access to the instrument parameters, which can then be saved as methods. Operational parameters can be optimized for specific analyses.

Sample ID

An operator-defined number between 0 and 9999, which automatically increases incrementally from the original for each sample (but not for replicates).

Sample Size

The size of the sample being analyzed. Sample size must be based on multiples of loop size. For 1-mL loops, the range of sample size is 1–25 mL (in 1-mL increments). For 5-mL loops, sample sizes are 5, 10, 15, 20, or 25 mL. For 10-mL loops, sample sizes are 10 or 20 mL. For 25-mL loops, the only sample size is 25 mL.



Loop Size

Allows the operator to set the current size of the loops installed in the Model 1010.

Note: This is not the actual volume of the sample loop since loop volumes are calibrated on the Model 1010 and variances in loop volumes, tubing lengths, and valve internal volumes will affect the actual loop volume. The true loop volumes are stored in memory so that when a sample size is entered, the actual sample volume is established based on true loop volume. The true sample volume will be on the printout when the results of the analysis are printed.

REAGENT VOLUMES Acid

Allows the volume to be set between 0 μL and 2000 μL in 100- μL increments.

Oxidant

Allows the volume to be set between 0 μL and 8000 μL in 100- μL increments.

TIME

Allows the operator to set the times used for analysis.

TIC React

Allows the operator to set the time that the phosphoric acid reagent converts TIC to carbon dioxide. Range of time is 00:01–10:00 min.

TIC Detect

Allows the operator to set the time that the carbon dioxide from TIC is purged from the sample. Range of time is 00:45–10:00 min.

TOC React

Allows the operator to set the time that the persulfate reagent converts TOC to carbon dioxide. Range of time is 00:01–30:00 min.

TOC Detect

Allows the operator to set the time that the carbon dioxide from TOC is purged from the sample. Range of time is 00:45–10:00 min.

TC React

Allows the operator to set the time that the acid and persulfate reagents convert TIC and TOC to carbon dioxide. Range of time is 00:01–30:00 min.

TC Detect

Allows the operator to set the time that the carbon dioxide from TIC and TOC is purged from the sample. Range of time is 00:45–10:00 min.

Auto Repeat

Allows the operator to set the time to delay the sample of the Model 1010. Range of time is 00:00:00–24:00:00 min (0 to 24 hours).



RINSE

Allows the operator to program rinses (0 to 50) to occur between replicates of the sample (per rep) or between samples (per sample). If the **Rinse Volume** and **Per Sample** are 0, no rinses will occur.

Note: If rinses are programmed per rep and per sample, only the rinse per sample will occur after the last repetition.

Control Keys

Files are managed by pressing [L] for **LOAD**, [S] for **SAVE**, or [D] for **DELETE**. Pressing any of these keys will open a block within the screen that lists the ten methods that are currently stored. The Up [↑] or Down [↓] arrow keys are used to select the method and [Enter] is used to activate the function once the method is selected. The operator can save a method using an alphanumeric name up to 15 characters.

Note: Loading, saving, and deleting files cannot be performed during analysis.

SPL MODE

Allows the operator to select the analysis mode for samples by using the [Page Up] or [Page Down] keys. The mode displayed is the current analysis mode. Choices include **TC**, **TIC**, **TOC**, and **TIC/TOC**.

STD MODE

Allows the operator to select the analysis mode for standards and check standards by using the [Page Up] or [Page Down] keys. The mode displayed is the current analysis mode. Choices include **TC**, **TIC**, and **TOC**.

Total Time

Displays the amount of time required to complete a run.

Note: Consistent times should be used for standards, samples, and check standards to ensure accurate, repeatable analyses.

SAMPLE INTRODUCTION

Displays the sample introduction method. The [Page Up] or [Page Down] keys toggle to choose from **Sipper**, **Autosampler**, **Syringe**, and **On-Line**.

Sipper

Allows samples to be introduced through the sipper tube from a sample bottle. If the TOC Analyzer is performing multiple sample or standard analyses, the unit will indicate when it is ready for the next sample. Use Table 4.3 to select the correct sample volume according to sample carbon concentration.



Table 4.3. Approximate Detectable Range for Sample Volumes (RSD values will vary with sample volume and concentration)

Sample Carbon Concentration	Sample Loop Volume
2 –20 ppbC*	25 mL
20 ppbC–5 ppmC	25 mL
100 ppbC–12.5 ppmC	10 mL
200 ppbC–25 ppmC	5 mL
1–125 ppmC	1 mL

*Concentrations below 20 ppbC are typically analyzed on-line, not using grab sample techniques

Note: For best results, select the sample loop volume so that the sample range falls in the middle of the concentration range of the sample loop.

Samples with carbon concentrations up to 125 ppmC can be analyzed with the 1-mL loop sampling capability. Samples with concentrations greater than 125 ppmC should either be diluted or injected by syringe.

Autosampler


Allows the operator to configure the Model 1051 Autosampler. For information on autosampler configuration, refer to the *Model 1051 Autosampler Operator's Manual*.

Syringe

Allows the operator to inject samples when prompted on the screen. The NDIR is linearized over a range of 0 to 125 µgC. A syringe injection volume appropriate to introduce this range of carbon per sample should be selected, depending on the sample's carbon concentration. Use Table 4.4 to select the proper sample injection volume.

Table 4.4. Approximate Detectable Range for Sample Injections (RSD values will vary with operator technique and syringe versus concentration)

Sample Carbon Concentration	Syringe Injection Volume
2 –200 ppmC	0.5 mL
5 –500 ppmC	0.2 mL
10 –1,000 ppmC	0.1 mL
20 –2,000 ppmC	50 µL
50 –5,000 ppmC	20 µL
100–12,500 ppmC	10 µL


CAUTION:
 When sample loops are changed from one size to another, be sure to change the loop size on the CONFIGURATION SCREEN [F5] and any sample standard, or rinse volumes that may be affected by a loop size change.



CAUTION:
*If Printer Enable
is disabled,
information will
be lost since the
Model 1010 does
not store data.*

	<p>Note: The maximum syringe injection volume is 1.0 mL. For larger volumes, use one of the loop-based injection modes.</p>
On-Line	<p>Configures the software for an on-line or process application. Water may be sampled from flowing processes if the sample loop is plumbed on-line with the process water. If the process water pressure is sufficient (5 psi or greater) the water pressure can be used to flow sample continually through the sample loop. In the case of pressure-fed flow through (on-line) sampling, the sample pump downstream of the loop must be bypassed to avoid creating back pressure from the flow-line. This is accomplished by connecting (finger-tight) the fittings together that are normally connected to the pump inlet and outlet.</p>
PRINTER	<p>Allows control of the printer that is connected to the TOC Analyzer.</p>
Printer Enable	<p>Turns on printer output so information can be printed.</p> <p>Note: Data is not saved on the TOC diskette and must be printed to retain a record.</p>
Print Method	<p>Enables printing of the method as it currently exists in the TOC Analyzer.</p>
Print Statistics	<p>Enables printing of the replicate average and standard deviation of samples and standards.</p>
ALARMS	<p>Allows the Model 1010 to provide a visual warning when TC, TOC, or TIC high or low concentration limits are exceeded. The Model 1010 activates relay closures through the alarm relay board for external warning devices. Values can be entered from 0.000 to 10,000 ppmC.</p>
CARBON MASS ALARMS	<p>Allows the operator to enable or disable the carbon mass overrange warning, which for the Model 1010, is activated at 130 µgC.</p>
CALIBRATION	<p>Allows the editing of standard calibration by permitting an entire standard to be deleted. If Allow editing? is OFF, standards may not be deleted.</p>
Date	<p>Displays the current date. This field may be edited for correction. When entering time values, it is only necessary to enter the numbers.</p>



Time

Displays the current time. This field may be edited for correction. When entering time values, it is only necessary to enter the numbers.

CALIBRATION RESULTS SCREEN

When [F6] is pressed, the CALIBRATION RESULTS SCREEN (CALIB SCR) appears.

The CALIBRATION RESULTS SCREEN displays the results of the current calibration and allows the operator to delete standards. The TOC Analyzer can be calibrated on one to five points. In order to simplify this process, the TOC Analyzer will compute a calibration curve after each standard is analyzed. This allows the operator to view the **Response Factor** (RF), and the coefficient of correlation **R²** (R²) for the calibration line as it is being “built.” The Model 1010 uses a least-squares fit method of calibration based on the number of standards used to calibrate the unit.

RUN STATUS		TOC 1010 Version X.XX			UNIT STATUS	
Stopped					System Ready	
CALIBRATION RESULTS						
STANDARDS TOC				STANDARD ANALYSIS [STD#1]		
STD#1	000.00000	ppm	010 ml	#	STD	Conc
STD#2	000.50000	ppm	010 ml			Vol
STD#3	001.00000	ppm	010 ml			Area
STD#4	005.00000	ppm	010 ml	Average = 0.00000		
STD#5	010.00000	ppm	010 ml	Standard Dev = 0.00000		
				Rel Std Dev = 0.00000		
Response Factor = 1.15600 ugC/k-cnt						
R ² = 0.000						
F1	F2	F3	F4	F5	F6	F7
START	HOLD	RUN SCRN	SEQ SCRN	CONFIG SCRN	CALIB SCRN	DIAG SCRN

STANDARDS

Displays current standards that are programmed into the TOC Analyzer and marks the standards that are used for the current calibration with “Used.”

Response Factor

Displays the response factor.

R²

Displays the coefficient of correlation for the calibration line as it is being “built.”

STANDARD ANALYSIS Displays an average, a standard deviation, and a relative standard deviation. To scroll through the current standard results, press [Page Up] or [Page Down]. To delete a standard from a calibration (if **Allow Editing** is turned on from the CONFIGURATION SCREEN), scroll through the standards until



the standard to be deleted is displayed on the monitor and press [Delete].

Note: The TOC Analyzer will recalibrate on the remaining standards and calculate new RF and R² values. If all standards are deleted, the TOC Analyzer will use a default calibration Response Factor of 1.156 µgC/1000 counts.

DIAGNOSTICS SCREEN

When [F7] is pressed, the DIAGNOSTICS SCREEN (DIAG SCR) appears.

The DIAGNOSTICS SCREEN provides tools for troubleshooting the TOC Analyzer by displaying information, including the current status and current readings, and allowing the operator to manually control the various mechanical components. Manual operation should be performed with the instrument in the Standby State.

RUN STATUS		TOC 1010 V5.2		UNIT STATUS					
Stopped				Low Gas Pressure					
DIAGNOSTICS									
STATE: STANDBY		[L] LEAK CHECK		ANALOG CONCENTRATION SIGNAL					
PRIME REAGENTS		[C] CALIBRATE LOOP		In TIC/TOC mode,					
Acid	0 times	[D] MANUAL DRAIN		signal shows TIC					
Oxidant	0 times	SAMPLE LOOPS		SIGNAL RANGE					
Pre Acid	0 times	Current Loop Size 5 ml		Min Conc (ppm C) 0.000					
ACTUATE		Loop A B		Max Conc (ppm C) 125.000					
Sample Pump	OFF	1ml	1.000	1.000	Use Timer No				
Drain Valve	OFF	5ml	5.000	5.000	Timer Duration 00:01:00				
Reverse Valve	OFF	10ml	10.000	10.000	Test Signal OFF				
Rinse Valve	OFF	25ml	25.000	25.000	GAS SAVER				
Loop Valve	Loop A	CURRENT READINGS		Off Type Never					
Transfer Valve	OFF	Block Temp 0.0 C		Off Time 18 : 59					
IR Valve	OFF	Sample Temp 0.0 C		On Type Never					
Cooling Fan	OFF	Pressure 0.0 psi		On Time 07 : 01					
Needle Wash Valve	OFF	Signal 0		Cleanup Blanks 6					
Pre-Purge Valve	OFF			Date 04 / 07 / 2003					
Septum Valve	OFF			Time 12:37					
F1	F2	F3	F4	F5	F6	F7	F8	F9	F10
START	HOLD	RUN SCRN	SEQ SCRN	CONFIG SCRN	CALIB SCRN	DIAG SCRN	ERROR SCRN		SPIRAL TRAY

STATE

Displays the current mode of the TOC Analyzer.

PRIME REAGENTS

Allows the acid and oxidant pumps to be primed by entering the number of times the reagent pumps are to be activated. The range is 1–99. If the current sample introduction mode is Autosampler, the Model 1051 Autosampler rinse and preacid pumps may be similarly primed.



ACTUATE	Allows the operator to manually control the valves, cooling fan, and sample pump. The items in this section can be changed by pressing the [Page Up] or [Page Down] keys. The [Page Up] key turns the component on and the [Page Down] key turns the component off.
CALIBRATE LOOP	Aids in the calibration of sample loops. Press [C] and follow the instructions on the screen.
MANUAL DRAIN	Allows the operator to manually drain the system.
Current Loop Size	Provides the same volume information as Sample Size on the CONFIGURATION SCREEN and STANDARDS on the SEQUENCE SCREEN. Can be changed by scrolling [Page Up] or [Page Down] through the choices.
CURRENT READINGS	Includes the sample temperature, block temperature, pressure, and NDIR signal. The NDIR signal is displayed in area counts.
ANALOG CONCENTRATION SIGNAL	Sends the sample concentration result as either a 4–20 mA or 0–10 V analog signal to a device such as a chart recorder. The signal can be either TIC or TOC mode. The Min Conc and Max Conc ranges define the lower and upper limits of the concentration within the 4–20 mA or 0–10 V signal. Program the signal duration through the timer duration input. The timer duration format is hr:min:sec.
GAS SAVER	Allows the operator to specify conditions to automatically turn OFF or ON the nitrogen flow to the Model 1010. Set the conditions to turn the gas OFF in either inactivity time (hrs) or time of day (24-hr time format). Turn the gas ON either every day (Monday–Sunday) or only on weekdays (Monday–Friday). For example, the following settings turn the nitrogen OFF at 5:00 PM and turn the nitrogen ON at 7:30 AM on Monday morning. OFF Type: Time of Day OFF Time: 17:00 ON Type: Weekdays ON Time: 07:30



Chapter 5 Operation



CAUTION:
Operating the keyboard and monitor while using WinTOC will cause the system to fail. Use either WinTOC or the keyboard and monitor but not both.

The OI Analytical Model 1010 TOC Analyzer can be controlled from either the keyboard using the control software provided or from a host computer using the WinTOC software. This chapter provides outlines the procedures for the operation of the Model 1010 using the control software (“Firmware”). For a complete list of commands, refer to Chapter 4, “Introduction to Firmware.”

Note: To operate the TOC Analyzer using WinTOC, see the *WinTOC 1010 Operator’s Manual*.

Overview

A water sample may be introduced into the Model 1010 via syringe injection or a pair of calibrated sample loops. Once the sample has been introduced, the entire analysis sequence is automatic.

Sample Introduction

The sample introduction step is performed via syringe injection or sample loops. The sample loops afford greater consistency of injection volume, whereas the syringe injection port allows sample injection of microliter quantities, with extremely high carbon concentration.

The multi-loop (Dual Loop Fluid Injector) capability uses two loops of identical volume. The pairs of loops can be 1 mL, 5 mL, 10 mL, or 25 mL. When the sample volume is a multiple of the loop volume, one loop will fill, then the sample valve will rotate allowing the first loop to empty into the digestion vessel, while the second sample loop fills. The sample valve will rotate again and empty the second loop while the first loop fills again. This continues until the sample volume is reached.

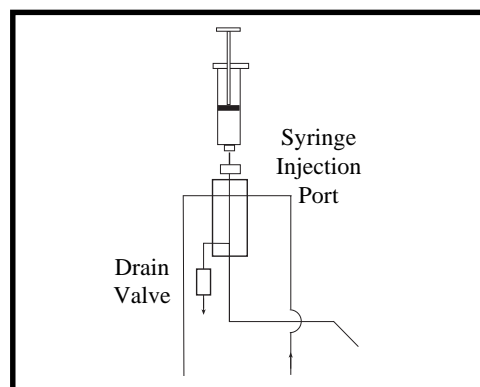


Figure 4.1. Sample Inject Step by Syringe

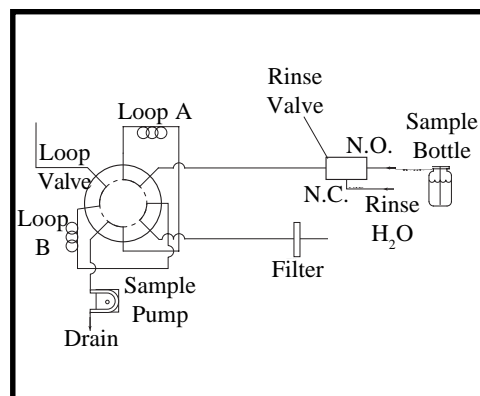


Figure 4.2. Sample Inject Step by Sample Loops



TIC React

Sample introduction is followed by adding a metered amount of acid reagent into the digestion vessel. TIC is released as carbon dioxide, which accumulates in the sample in the digestion vessel.

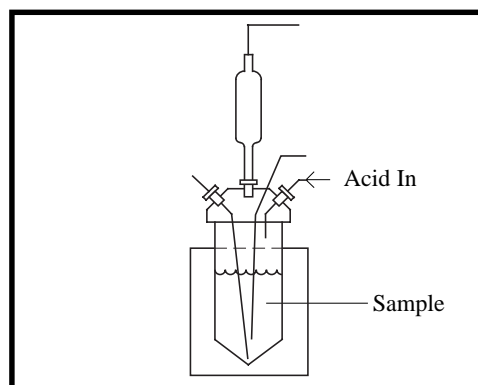


Figure 4.3. TIC React Step

TIC Detect

After a preset reaction time, the digestion vessel is placed in-line with the NDIR, and a gas stream purges out any carbon dioxide formed from inorganic carbon in the sample. This carbon dioxide is carried to an NDIR where it is detected. The NDIR has been calibrated to directly display the mass of carbon dioxide detected.

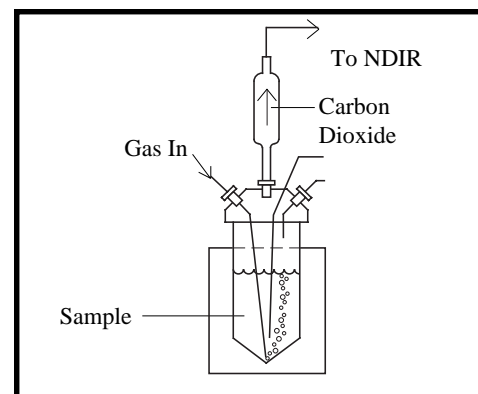


Figure 4.4. TIC Detect Step

TOC React

Purge gas flow to the digestion vessel is stopped and a metered amount of sodium persulfate reagent is added to the sample. As the temperature is increased to 100°C, the persulfate reacts with organic carbon in the sample to produce carbon dioxide, which accumulates in the sample in the digestion vessel.

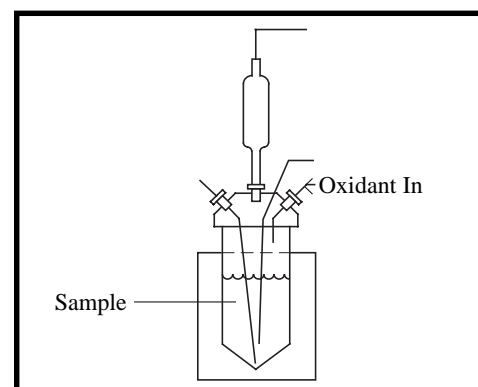


Figure 4.5. TOC React Step

TOC Detect

After a preset reaction time, the digestion vessel is placed in-line with the NDIR, and a gas stream purges out any carbon dioxide produced by the persulfate oxidation. This carbon dioxide is carried to the NDIR where it is detected. The NDIR has been calibrated to directly display the mass of carbon dioxide detected.

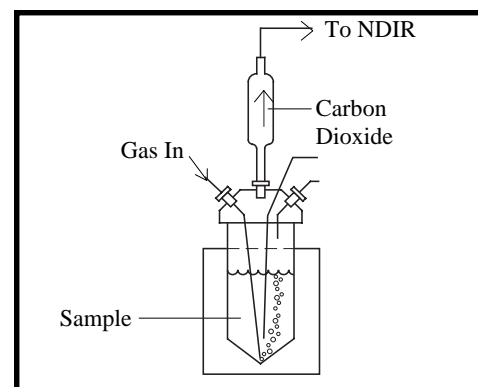


Figure 4.6. TOC Detect Step



TC React

A metered amount of sodium persulfate reagent and phosphoric acid reagent are added to the sample. As the temperature is increased to 100°C, TIC is released as carbon dioxide and persulfate react with organic carbon in the sample to produce carbon dioxide. This carbon dioxide accumulates in the sample in the digestion vessel.

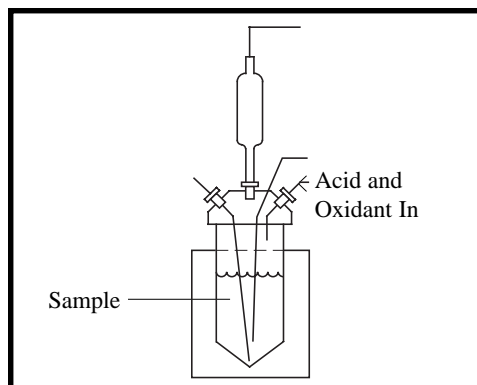


Figure 4.7. TC React Step

TC Detect

After a preset reaction time, the digestion vessel is placed in-line with the NDIR, and a gas stream purges out any carbon dioxide produced. This carbon dioxide is carried to the NDIR where it is detected. The NDIR has been calibrated to directly display the mass of carbon dioxide detected.

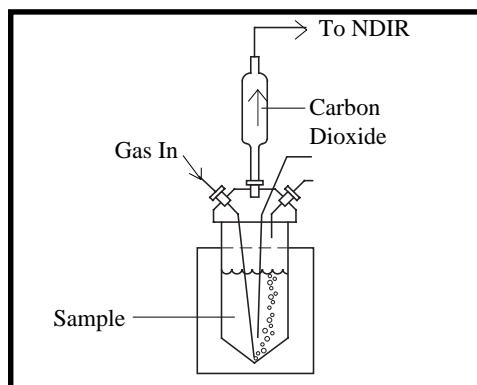


Figure 4.8. TC Detect Step

Drain Step

The gas flow in the digestion chamber is reversed and the sample waste is carried out of the chamber to drain.

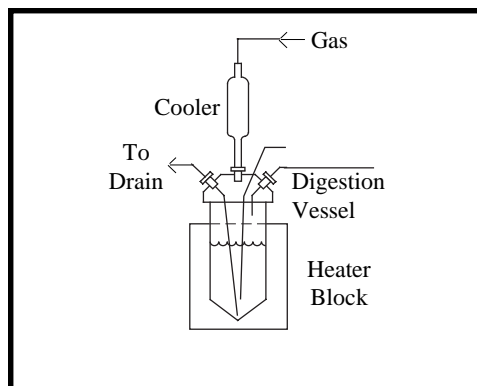


Figure 4.9. Drain Step

Rinse Step (Optional)

Rinses can be programmed after replicate analyses or between samples. After the drain cycle, rinse water is pumped and transferred through the sample pathway to remove any trace of the previous analysis. Once the rinse cycle is complete, the rinse water is drained.

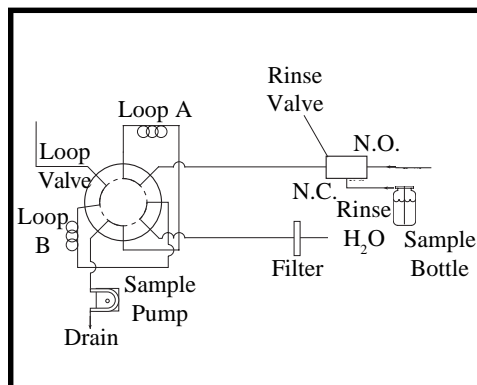


Figure 4.10. Rinse Step



Keyboard and Monitor Operation

In this configuration, a PC-type keyboard is used to control and operate the Model 1010. To control the Model 1010 with WinTOC software, refer to the *WinTOC 1010 Operator's Manual*. The descriptions below refer to operation of the Model 1010 using a keyboard and monitor.

The Model 1010 is controlled by entering keystrokes on the keyboard. The monitor allows the operator to view the input and the results of these keystrokes. To simplify operation, all operations and functions can be viewed from five screens. The five screens are as follows:

- RUN SCREEN [F3]
- SEQUENCE SCREEN [F4]
- CONFIGURATION RESULTS SCREEN [F5]
- CALIBRATION SCREEN [F6]
- DIAGNOSTICS SCREEN [F7]

Table 5.1 lists the primary keys and their functions.

For more information on these screens, see Chapter 4, "Introduction to Firmware."

Table 5.1. Description of Key Functions

Key	Function
[F1]	Start/resume an analysis
[F2] (pressed once)	Pause/drain an analysis
[F2] (pressed twice)	Stop an analysis
[F3] through [F7]	Move between screens
[F8]	Display error screen
[F10]	Spiral the autosampler tray
[↑] or [↓]	Move within a screen
[Page Up]	ON or toggle view
[Page Down]	OFF or toggle view
[Esc]	Exit current function, saving any entered values



Starting up the Unit

Powering up the Model 1010 loads the system program from the processor board memory chip (chip disk).

1. Ensure that there is **no** floppy disk in the Model 1010 disk drive. The disk drive is only used for initial program installation at the factory and for system program upgrades.
2. Turn on the power switch.
3. During the Model 1010 boot-up, listen for a series of audible beeps to determine the status of the instrument. The beep sequence is:
 - 1 Beep - System startup
 - 2 Beeps - CMOS check passed
 - 3 Beeps - Firmware ready

Note: The audible beeps will not be heard on Model 1010 Rev. A, B, C, D, or E Analyzers. To modify a Model 1010 for this function, contact the OI Analytical Technical Support Department. If the Model 1010 does not perform the beep function, the floppy disk light can be used as a guide. This light will cycle two times—the first time loads the operating system (»25 sec) and the second time loads the program (»25 sec).

Analyzing Reagent Blanks

Theory

If a complete analysis sequence is performed (i.e., REACT/DETECT) without injection of a sample, the detector responses for TIC and TOC will still be generated due to carbon in the reagents, gas, tubing, and digestion vessel. These reagent blanks can be reduced to a minimum with consistent values but cannot be completely eliminated.

When standards, samples, or check standards are analyzed, it is assumed that the detector response generated from the analysis includes response due to the reagent blank in addition to the carbon in the sample. If the reagent blanks for TIC, TOC, and POC are determined prior to the analysis of standards, samples, or check standards, the blank carbon mass may be subtracted from the sample or check standard carbon mass to accurately determine the amount of carbon due only to the sample.

Run reagent blanks until replicate values are consistent prior to sample analysis, using the same conditions of analysis as planned for the samples. Conditions of analysis are generally constant for routine samples, but time and volume parameters may vary.

Note: The Model 1010 automatically enters a rolling average of the last three reagent blank values run on the unit.



Procedure

1. Turn on the Model 1010.
2. If any current settings for volumes and times are not correct for the analysis to be performed, set the new conditions of analysis on the CONFIGURATION SCREEN [F5].
3. Press [F4] to select the SEQUENCE SCREEN.
4. Select [1] for **Reagent Blank**.
5. Enter the number of reagent blanks to be analyzed (0 to 999) and press [ENTER].
6. Press [F1] START. The Model 1010 will begin analyzing blanks, and will provide a screen display showing the status of the blanks and of the current blank being analyzed.

(To hold the run, press [F2]. The run can then be resumed by pressing [F1] or aborted by pressing [F2] again).

Note: The printed results from the printer for the reagent blanks are reported in integrator counts. Typically, IC blanks should be less than 200 counts. OC blanks should be less than 200 counts for 100 g/L sodium persulfate reagent and less than 300 counts for 200 g/L sodium persulfate reagent. Stable blanks are within ± 50 counts. Reagent blank values will vary depending on the purity of water and the quality of the chemical reagents used.

Note: Stable blanks should be established prior to standard, sample, or check standard analysis. If blank values are above the typical range, this is due to high water content or the quality of the reagent being used. If the reagent blanks are high, but stable (within ± 50 counts), the instrument will still provide satisfactory performance.

Calibration

Theory

The wet oxidation method for TOC requires reagent solutions (reagents) of phosphoric acid and sodium persulfate to be added to standard solutions (standards) and samples. Reagent water and reagent materials are needed for preparing reagents, while reagent water and standard materials are needed for preparing standards. All of the materials involved, including reagent water, reagent materials, standard materials, and samples, contain organic carbon (see Figure 4.9). The organic carbon in the reagent water and in the reagent materials is not wanted but impossible to avoid. The organic carbon in the standard materials is known (as a percentage of the total mass of material to be added) and is typically varied to produce a set of standards of differing concentrations of carbon. The organic carbon in

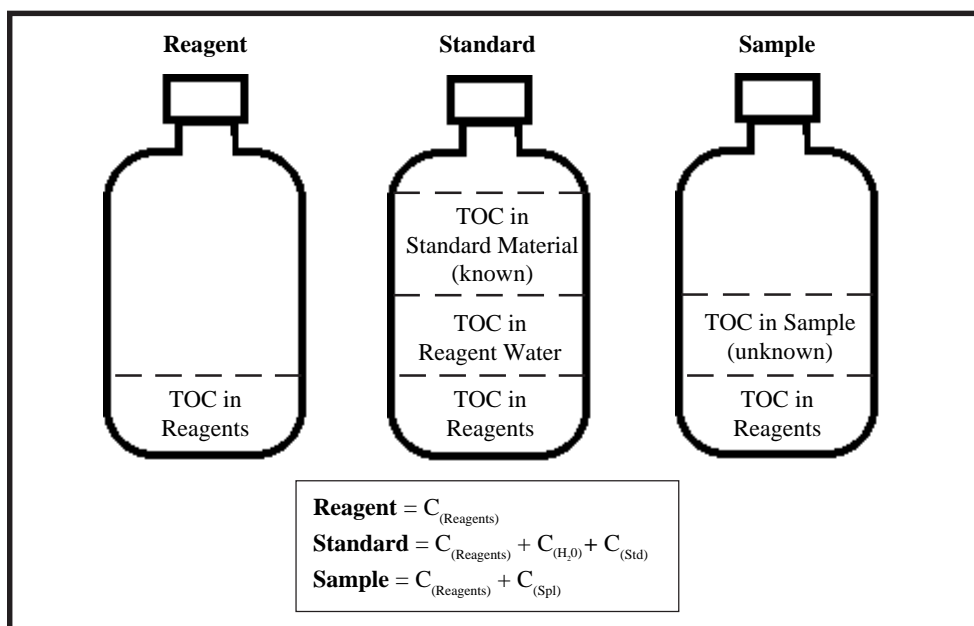


Figure 4.9. TOC in Reagent, Standard, and Sample

the samples is unknown but can be determined by comparison of the standards' analysis results if the carbon from all sources is properly considered.

When standards are analyzed, organic carbon from the following sources contributes to the response of the TOC Analyzer's NDIR detector: (1) reagent water used to make the standards, (2) reagents of acid and persulfate, and (3) standard material added to the standards. The contribution from (1) and (2) are presumably constant even when (3) purposefully differs with each standard. Therefore, the standards do not contain the carbon mass or concentration as labeled. Because of this, a method of standards additions is used for the calibration routine. Reagent water representing a standard of concentration zero-added can be run as one of the standards.

The mass of carbon added to each standard (in μgC) is calculated from net (prepared) concentrations and volumes. A least-squares linear regression is performed on mass:area (X:Y) pairs. From the slope of the regression line only (not using any intercept because of the standards additions approach), a response factor is calculated as micrograms carbon (μgC) per thousand area counts. A correlation coefficient is also calculated. This calibration approach eliminates the need to know the exact mass of the background carbon from the reagent water and reagents. The response factor representing the trend of detector response, with increasing carbon added, should be linear over the desired range (see Figure 4.10).

The carbon masses contributed by reagent blanks (BLK), which are produced from analyses of reagents only, are determined by multiplying their corresponding area counts by the response factor. The average of the latest three reagent blank masses (in μgC) is saved in memory. This Avg BLK Mass is updated as a rolling (last-three) average, as new blanks are run.

Each new sample and check standard area count produced from subsequent runs is multiplied by the response factor without subtracting any reagent blank

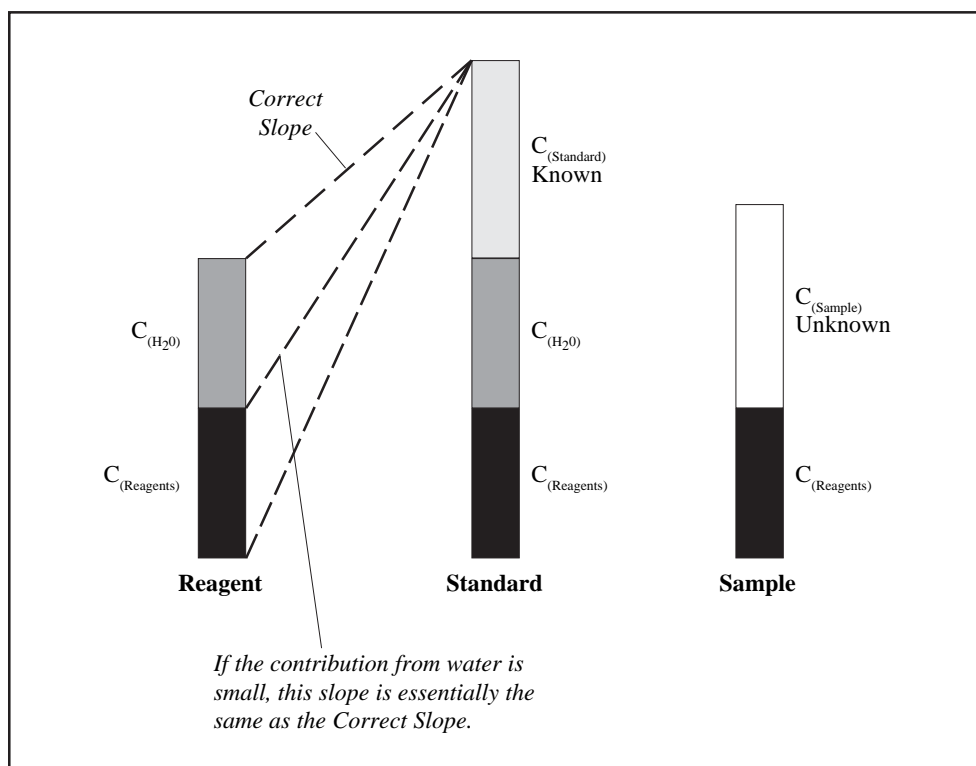


Figure 4.10. Carbon Content in Reagent, Standard, and Sample

area. This will produce a gross carbon mass measurement (in μgC) for each run. Because reagents were added to samples and check standards for their analysis, the Avg BLK Mass is subtracted from each sample and check standard mass measured. The concentration of each sample and check standard is then calculated by dividing its reagent blank-corrected mass by its run volume. Check standards have contributions of carbon from the same sources as regular standards (they could actually be the same solutions) so the unwanted carbon from the reagent water must also be subtracted to yield the added carbon only, rather than carbon in the standard from all sources. The carbon mass from the reagent water is determined by using the Y-intercept point from the regression line above. This Y-intercept represents the area counts from reagent water only. If water is run as one of the standards, this predicted value will be much more accurate. This water-contribution area response is converted to mass with the response factor and then subtracted. In this way, the results from check standards will yield only the added carbon, not the total carbon (including the unwanted carbon) in the solution.

As mentioned earlier, running the reagent water as a zero-concentration standard will adjust the Y-axis value on the calibration curve and provide the correct water value to subtract from the check standard, to arrive at the correct standard value (see Figure 4.11).

To illustrate the effects of the water value on the calibration, first the instrument is calibrated with water and a 500 ppbC standard. Then, the same 500 ppbC standard is run as a sample. The value that is returned will be higher than the 500 ppbC value because the water in the standard is not taken into account. Samples in the Model 1010 have only the reagent blank value subtracted. If the same

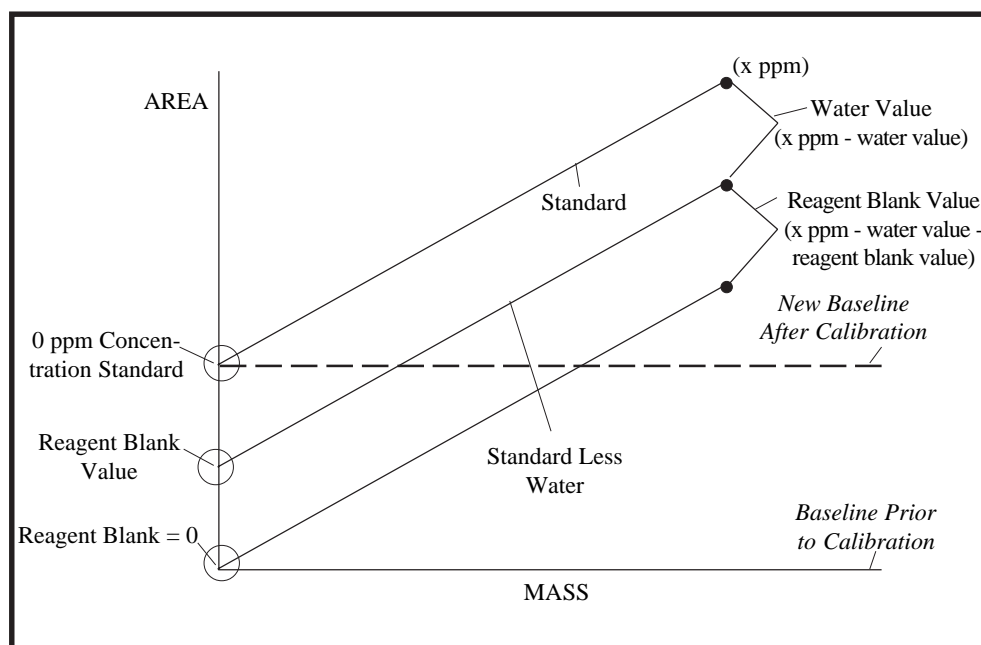


Figure 4.11. TOC Calibration Curve

500 ppbC standard is run as a check standard, the true value of 500 ppbC is returned because check standards have the reagent blank value and the water value subtracted (by using the Y-intercept offset). In a single-point calibration, the reagent blank value would be used as the second point in the calibration. In this case, the check standard value may not agree with the true standard value, as the unit assumes that the water value is negligible or very close to the reagent blank value. Therefore, at the low levels of analysis, it is recommended that water is analyzed as a zero-concentration standard to determine the exact value of the water. (If the operator does not run water, the Model 1010 will automatically calculate a water value.) If the water value is 100 ppbC and the standard is 5.0 ppmC, the total standard concentration is 5.1 ppmC. However, if the standard is 100 ppbC, the total standard concentration is 200 ppbC. At the lower level, knowing the exact value of the water is crucial.

For best results, follow these guidelines:

- No matter how many points are used for calibration, one of the points should be beyond the highest sample to be analyzed.
- For low-level work, water should be analyzed as a zero-concentration standard.
- For any range of samples, but especially at the lower levels, run as few standard points as necessary. Running a minimum amount of standards lowers the risk of operator error. If any of the standards are made incorrectly, the calibration curve will be shifted due to the standard.
- Single-point calibration is not possible when using water as a single point.



TOC Method

Using Potassium Biphthalate (KHP)

For calibration using organic carbon (TOC), known volumes (up to five) of a solution of potassium biphthalate are introduced. One recommended calibration (stock) solution is 1000 ppmC in distilled or deionized water. Each microliter of this solution contains 1.0 microgram of carbon so that the introduction of a specific number of micrograms of carbon is accomplished by introduction of the same number of microliters of solution. The number of micrograms of carbon introduced should be similar to the mass of carbon expected for samples. For low-level analyses, a calibration solution of 100 ppmC, 1000 ppbC, or 100 ppbC may be more suitable so that larger volumes may be introduced for better precision.

Standard solutions have a shelf life of three weeks.

Constant Volume Procedure

The routine calibration methods typically call for injections of 5 to 50 μL of standard solution, whereas sample volumes of 0.5 to 10 mL are used for all but high TOC samples (>100 ppmC). If calibration using the same volume of standard used for samples is desired, a standard solution of carbon concentration similar to the samples may be prepared (using TIC or TOC). Calibration in this manner allows unattended replicates of the standard to be run, using the sample loop. Either the TIC or TOC calibration mode can be used in this manner, depending on the nature of the standard material.

TIC Method

Using Sodium Carbonate

For calibration using inorganic carbon (TIC), known volumes (up to five) of a solution of sodium carbonate are introduced. One recommended calibration solution is 1000 ppmC in distilled or deionized water. Each microliter of this solution contains 1.0 microgram of carbon so that the introduction of a specific number of micrograms of carbon is accomplished by introduction of the same number of microliters of solution. The number of micrograms of carbon introduced should be similar to the mass of carbon expected for samples. For low-level analyses, a calibration solution of 100 ppmC may be more suitable so that larger volumes may be introduced for better precision.

Solutions of sodium carbonate are basic and will absorb CO_2 from the atmosphere; they should remain sealed when not being sampled. Standard solutions have a shelf life of three weeks.

Calibration Procedures

Overview

After information is programmed, the calibration of the Model 1010 is totally automated. Standard parameters are programmed into memory for recall during calibration, a calibration sequence is programmed, then standards are analyzed. Once this analysis is complete, the Model 1010 is considered calibrated.



Calibration can be performed using one to five points. If one standard is to be run, the current blank average is used as the necessary second point for the calibration curve. This technique assumes the contribution from the reagent water to be negligible. As the Model 1010 runs standards, it recalibrates after completing each standard analysis. For example, after the first standard has been run, the Model 1010 calibrates based on that standard and the current blank average.

Note: The Model 1010 will “overwrite” a standard in memory each time a new calibration is run and that standard is analyzed. For example, if three reps of standards #1, 2, and 3 were used to calibrate the Model 1010 yesterday and the same three standards (#1, 2, and 3) are used today, today’s results will overwrite yesterday’s. However, if standards #1, 2, and 4 were used yesterday and standards #1, 2, and 3 are used today, #4 will remain in memory. To remove standard #4 from memory, see “**CALIBRATION SCREEN**” in this chapter.

Note: If no calibration curve exists and water is analyzed as the first calibration standard, the response factor and the standard curve will be useless. This is because the Model 1010 is using the blank value and water value for its two calibration points. After the second standard, the Model 1010 will drop the blank value as the necessary second calibration point and use the second standard point. The Model 1010 will “build” the calibration curve as more standards are analyzed.

Programming Standard Information

1. Press [F5] for the CONFIGURATION SCREEN.
2. Use the Up [↑] or Down [↓] arrow keys to move the cursor to the **STD Mode** location.
3. To change the standard mode use the [Page Up] or [Page Down] keys. The choices are **TOC**, **TIC**, or **TC**.
4. Press [F4] for the SEQUENCE SCREEN.
5. Modify the standards as necessary by moving to the information to be changed with the Up [↑] or Down [↓] arrow keys and changing the information using the numeric keys.
6. Press [Esc] to exit the **Standard** section of the SEQUENCE SCREEN.

Running a Calibration Sequence

1. Press [F5] for the CONFIGURATION SCREEN.
2. Change the **STD Mode** if desired
3. Press [F4] for the SEQUENCE SCREEN.



4. Press [2] to select **Standard**.
5. Enter the desired standard number (STD #1–5) from the standards programmed into memory and press [ENTER].
6. Enter the number of replicates to be run of that particular standard and press [ENTER].
7. Repeat the above steps until all standards to be run are entered into the sequence.
8. Press [F1] to start the calibration sequence.
(To hold the run, press [F2]. The run can then be resumed by pressing [F1] or aborted by pressing [F2] again).

Note: While the Model 1010 is not running, calibration sequences as well as other sequences can be saved by pressing [S] **SAVE** after programming the sequence (and while the SEQUENCE SCREEN is displayed).

Note: Pressing [Esc] will delete the current line being entered in the **SEQUENCE TABLE**.

Note: The response factor for an acceptable calibration should be in the range of 0.9–1.5.

Note: Calibration of the Model 1010 cannot be performed with volumes less than 1 mL unless the unit is calibrated in the syringe mode. If syringe injection is used for the sample introduction mode, the volumes desired for injecting must be entered under **STANDARDS (STD #1–5)** on the SEQUENCE SCREEN [F4].

Note: The Model 1010 can analyze up to 10 replicates of a standard.

Running Check Standards

Running check standards allows the operator to run a known standard to check the current calibration of the Model 1010, without affecting the current calibration. Check standards values will have the reagent blank value and the water value subtracted. Check standards are recalled from the same standards as programmed on the SEQUENCE SCREEN.

1. Press [F4] for the SEQUENCE SCREEN.
2. Press [4] to select **Check Standard**.
3. Enter the standard number (**STD #1–5**) from the standards programmed into memory and press [ENTER].
4. Enter the number of replicates to be run of that particular check standard and press [ENTER].



5. Repeat the above steps until all check standards to be run are entered into the sequence.
6. Press [F1] to start the check standard sequence.

Note: While the Model 1010 is not running, calibration sequences as well as other sequences can be saved by pressing [S] **SAVE** after programming the sequence (and while the SEQUENCE SCREEN is displayed).

Running Samples

Running samples allows the operator to analyze an “unknown” and compare its response to known (standards) values. Sample values have the reagent blank values subtracted. Run samples by following these steps:

1. Press [F4] for the SEQUENCE SCREEN.
2. Press [3] to select **Sample**.
3. Enter the number of samples to be analyzed and press [ENTER].
4. Enter the number of replicates to be run of each sample and press [ENTER].
5. Press [F1] to start the sample analysis.
(To hold the run, press [F2]. The run can then be resumed by pressing [F1] or aborted by pressing [F2] again).

If differing numbers of sample replicates are to be analyzed, the samples with differing replicates can be entered into the sequence table as separate entry lines.

Note: While the Model 1010 is not running, this sequence can be saved by pressing [S] **SAVE** after programming the sequence (and while the SEQUENCE SCREEN is displayed).

Note: Pressing [Esc] will delete the current line being entered in the **sequence table**.

Sequencing

Sequencing allows the operator to program and run combinations of blanks, samples, standards, and check standards. To build a sequence:

1. Press [F4] for the SEQUENCE SCREEN.
2. Press the appropriate key ([1], [2], [3], or [4]) to select **Blanks**, **Standard**, **Sample**, or **Check Standard**, respectively.
3. Follow the steps above to program the sequence.



4. Repeat until the sequence is programmed.
5. Press [F1] to start the sample analysis.

If a mistake is made during programming, press [E] **Edit Table** to allow editing of the table to correct errors, or press [C] **Clear Table** to clear the table.

Note: While the Model 1010 is not running, this sequence can be saved by pressing [S] **SAVE** after programming the sequence (and while the SEQUENCE SCREEN is displayed).

Note: Pressing [Esc] will delete the current line being entered in the **sequence table**.

Files

Method

Up to ten methods can be saved from the CONFIGURATION SCREEN. This screen contains [L] **LOAD**, [S] **SAVE**, and [D] **DELETE** functions. Methods are composed of:

- TIC React Time
- TIC Detect Time
- TOC React Time
- TOC Detect Time
- SPL Mode: TIC/TOC/TC
- STD Mode: TOC/TIC/TC
- Sample Volume
- Reagent Volumes
- Rinse Information
- Auto Repeat Time

These method parameters will be saved or loaded when files are saved or loaded under the CONFIGURATION SCREEN. The listing of methods will include file "0", which is the default method "DEF(AULT) METHOD" for TOC analysis. The parameters of this method should provide adequate analysis conditions for typical TOC analyses. The default parameters are:

- TIC React Time: 2:00
- TIC Detect Time: 1:00
- TOC React Time: 2:30
- TOC Detect Time: 1:30
- SPL Mode: TIC/TOC
- STD Mode: TOC
- Sample Size: 10 mL
- Reagent Volumes: Acid 200 µL
Oxidant 1000 µL
- Rinse Information: 25 mL 1 Per Rep 1 Per Sample
(No Rinses)



Note: Loading, saving, or deleting files cannot be performed during an analysis.

Sequence

Sequences can be saved from the SEQUENCE SCREEN. Sequences are composed of analysis types: blanks, standards, samples, and check standards. These parameters will be saved or loaded when files are saved or loaded under the SEQUENCE SCREEN.

Analytical Hints

Clean Water Cycling

When the Model 1010 is to be operated after a long period of nonoperation, cycle clean water through the unit as sample to help remove any contaminants from the analyzer. This can be performed by:

1. From the CONFIGURATION SCREEN [F5], enter **Sample Size** as 25 mL and **Sample Intro** as Sipper.
2. From the SEQUENCE SCREEN [F4], select **Sample** [3].
3. Enter 1 for quantity and 10 for reps.
4. Place sipper tube in vessel of clean water.
5. Press [F1] to start analysis.

Confirming Recoveries in Difficult Samples

The default analysis conditions set in the Model 1010 are times, temperatures, and volumes that have been established to analyze the majority of water samples typically tested in laboratories. However, in the case of complex sample matrices, these conditions may not be adequate to render an accurate value for carbon concentrations in a sample.

There are several methods for determining recovery efficiency on a sample. One method, Standards Addition, is probably the most common technique used. Another would involve using a smaller or larger sample size and verifying that concentration (recovery) is equivalent in both cases. The method described here is used to not only give information on method recoveries but when completed, sets the conditions for analysis on the sample matrix in question. In brief, it requires the operator to vary the instrument's analysis parameters until a maximum, stable value is obtained.

Three parameters can be adjusted to achieve optimum analysis conditions. These are (1) temperature of the digestion vessel, (2) volumes of reagents used, which are the phosphoric acid (TIC) and persulfate (TOC), and (3) times



allowed for purging inorganic carbon and conversion of organic carbon to CO₂. For the most part, the first two parameters seldom need adjusting, especially the 100°C set point for the digestion vessel, since lowering the temperature much below 100°C starts slowing down reaction rates, and elevating the temperature causes excessive steam generation which can result in problems downstream of the reaction vessel. Increasing the reagent volumes may be necessary if samples have a high pH, particulated inorganics, or if carryover from one sample to the next is suspected. So, for the majority of difficult samples (brines, acids, caustics, SOC, etc.) the parameters that achieve the most significant changes are the extended reaction parameters.

To confirm that the time parameters are optimum, the operator should choose the suspected, “most difficult” sample to work up the analysis conditions. Beginning with default analysis parameters, run this sample two to three times to establish a trend. Then extend the time function that is believed to be too low using 30 second to 1 minute increments. That is, if low TIC recoveries are suspected or if it is believed that inorganic carbon is being carried over into the TOC, then extend the react time. If TOC recoveries are lower than expected, or they are not reproducible, extend the react time.

Optimum analysis time has been achieved when extending the time parameter in question has no significant increase in NDIR detector response. This increased response can be monitored in two ways, either by monitoring the Model 1010 output printer, or by monitoring the peaks as they appear on the run screen [F3] on the monitor. Remember that changing the analysis conditions will have some effect on the blank value which will increase slightly with increased time or reagent values.

Once new analysis conditions have been determined on the “most difficult” samples, the instrument can be calibrated and the other “less difficult” samples can be analyzed using the analysis method established.

TOC Analysis of Difficult Samples

As mentioned earlier, the majority of samples can be analyzed with the default parameters in the Model 1010. However, analyzing difficult samples can be achieved successfully using various analysis techniques. Most analysis problems fall into one of three categories:

1. Samples with pH problems (basic samples).
2. Samples containing components that compete with the organics for persulfate.
3. Samples containing long chain or complex carbons.

Depending on the sample type and matrix, the parameters below should solve the majority of analytical problems.



Samples with pH problems (basic samples)

Basic samples with a pH of 9 or higher do not allow the complete conversion of TIC to carbon dioxide when the default value of 200 μL of acid reagent is added to the sample. In order for this conversion to occur, the pH of the sample must be 2 or lower. To correct this problem, additional acid reagent must be added to the sample. To test if the pH is at the correct level, perform a pH test on the sample after a metered amount of the acid reagent is added. This can be done by using a syringe to dispense (in 100- μL increments) the acid reagent in the amount of sample that will be analyzed and testing the resulting solution with pH paper. If the correct pH is not achieved, continue to add acid reagent (in 100- μL increments) until the desired pH is achieved. Once the correct pH is achieved, increase the acid reagent volume on the Model 1010 to the amount required to lower the pH.

Samples containing elements that compete with the organics for persulfate

Inorganic halides in samples compete with the organics for persulfate. The Model 1010 is able to analyze samples with up to 30 mg of chlorine without any modification. When samples contain over 30 mg of chlorine, additional persulfate reagent, increased TOC react time, and a Halide Scrubber Option are necessary. To test if maximum recovery of the sample is achieved, increase the TOC react time by one minute on successive analyses until maximum recovery is reached. This same technique can be used with the persulfate reagent by increasing the amount of persulfate reagent by 100 μL increments until maximum recovery occurs. Suggested optimum settings for halide-containing samples are:

Sample Volume	1 mL
Acid	400 μL
Oxidant	5000 μL (100 g/L sodium persulfate)
TOC React Time	8:30

Note: If more persulfate reagent or acid reagent is added and/or the TOC react time is increased, the instrument should have the same volume of persulfate reagent and/or the same times used during the running of blanks. This is to ensure that the correct blank value is used when the samples and check standards are analyzed.

Note: Instead of increasing the persulfate reagent volume, it is possible to increase the persulfate reagent concentration. Instead of using 100 g/L concentration, increase the concentration to 200 g/L.

Samples containing long chain or complex carbons

The analysis technique to improve recovery of these types of samples is to increase the TOC react time until maximum recovery is achieved. To test if maximum recovery of the sample is achieved, increase the react time by one minute on successive analyses until recovery does not improve.

Note: If the TOC react time is increased, then the instrument should have the same volume of persulfate reagent and/or the same times used during the running of blanks. This is to ensure that the correct blank value is used when the samples and check standards are analyzed.



Standard Additions

A commonly used method in calibrating instruments involves the addition of a known standard to an unknown. By analyzing the unknown sample and a standard added to the unknown, the instrument's detection of the sample can be calculated. The difference between the instrument's response due to the standard plus unknown and the standard's true value is then used to adjust (or calibrate) the instrument. After the instrument is calibrated to the corrected value, the unknown can be analyzed directly.

The standard addition calibration is often used when analyzing TOC in ultrapure water. The term TOC is used in this application to mean Total Oxidizable Carbon; that is, those organic species that can be oxidized to carbon dioxide (CO₂) by a given oxidation method. Analyzing the oxidizable species in ultrapure water bears special consideration due to the low concentration of these species and to the fact that this same water is used to set the baseline and make the standards. When dealing within the range of 0–1000 ppb, the calibration method is quite important. The standard addition method is able to meet this precision and accuracy.

Rinses Per Replicate and Rinses Per Sample

The use of rinsing during analysis is recommended to help improve the performance of the instrument and to help meet performance specifications. Rinses per replicate are highly recommended when sample volumes below 10 mL are used. This will help prevent the carryover of reagents between replicates. Rinses per sample are recommended to prevent carryover or cross-contamination between samples.



Chapter 6

Maintenance

This chapter discusses both the routine and nonscheduled maintenance of the Model 1010 Wet Oxidation TOC Analyzer, starting with some general information and a maintenance schedule.

Schedule for Routine Maintenance

The operator is encouraged to set up an instrument logbook to record instrument operation time and document periodic maintenance. This logbook can be used to record results of inspections and component replacement necessary for proper maintenance of the Model 1010.

For the most reliable performance of the Model 1010, and as a condition of the warranty, the following schedule of routine maintenance should be followed. (Scheduled hours refer to number of hours of operation.)

Maintenance Item	Schedule
Reagent reservoirs	as needed
Injection port septum	50–200 injections
Indicating drying tube	400 hours (or as needed)
NDIR zero	100 hours
Gas service	as needed
Sample pump	2,000 hours
Digestion vessel/condensation chamber	as needed
Permeation tube	2,000 hours (or as needed)



Reagent Reservoir

The volumes of reagents in the bottles on the side of the Model 1010 should be inspected at times according to the number of analyses and volumes of reagents used per sample. The reagent bottles hold 1 L each. Smaller bottles are available through OI Analytical. Reagents should be added to keep the bottles from being completely emptied. Operation of the reagent pumps without liquid is not recommended. See Chapter 1, "Introduction," for the preparation of reagents for addition to these bottles.

Injection Port Septum

Behind the injection port is a 5-mm Teflon-faced, silicone rubber septum. This septum is pierced when samples are injected by syringe. Depending on the quality of the syringe needle, this septum may need replacement after 50 to 200 piercings. A water droplet in the injection port hole during sample-draining (near the end of each run) is an indication of a leaky septum. Though a leaky septum is cause for replacement, no sample loss will occur during injection, so the septum does not need to be replaced until a convenient time. Water that seeps from the injection port in this manner is the spent sample normally drained to waste under gas pressure. Change the septum as follows:

1. Insert a syringe needle (2" long) through the injection port guide.
2. With the syringe needle in the injection port guide, unscrew the guide counterclockwise.
3. When the injection port guide clears the final threads of the injection port block, remove the syringe with the guide and septum on the needle.
4. Discard the spent septum and place a new septum on the syringe needle, centered with respect to the injection port guide and with the Teflon face away from the injection port guide.
5. Install the new septum into the injection port block by screwing the injection port guide clockwise into the injection block until it just becomes finger-tight.
6. Check the injection port for leaks.



CAUTION:
Do not over-tighten.

Tube End Fittings

The Model 1010 uses tube nuts and ferrules to interconnect the valves, digestion vessel, and various other connections in the Model 1010. Do not over-tighten these fittings. Overtightening will distort the ferrules and cause the tubing to be constricted. If a fitting continues to leak after tightening, remove the nut and ferrule, cleanly cut back the tubing approximately 0.25"–0.50", and install a new ferrule.



Making 1/8" and 1/16" Flare Fittings with TFE Tubing

Note: Flared fittings are only used in the NDIR detector and on older (pre 2001) TOC Analyzers. On current models, flare fittings have been replaced with nuts and ferrules.

A 40 W soldering iron (Part #168808) with stainless steel flaring tips is used to thermally flare Teflon tubes. A 1/8" tip (Part #169129) and 1/16" tip (Part #169137) are required. Directions for flaring tubing are as follows.

1. Cut the tube end to be flared squarely at 90°.
2. Slide the appropriate tube end fitting and washer onto the tube. Allow 1/2" between the washer and the end of the tube to avoid preheating the washer.
3. Use a Kimwipe or sandpaper to grip the head of the tube end fitting and tubing. Press the tubing over the tip of the soldering iron. Apply pressure until the tip of the tubing flares out.
4. Slide the end fitting and washer firmly against the flare. Hold for approximately 1/2 second, immediately remove and press the flare against a flat, cool surface.
5. Inspect the flare for uniformity, diameter, and cracks or other defects. Tubing that is 1/8" should flare to a diameter slightly larger than the stainless steel washer. Tubing that is 1/16" should flare to approximately 1/8" diameter to hold the washer.

Definitions

1/8" TFE Tubing

1/8" O.D. x 0.062" I.D. TFE Teflon tubing (Part #147901).

1/16" TFE Tubing

1/16" O.D. x 0.031" I.D. TFE Teflon tubing (Part #145591).

1/8" Tube End Fitting

Tube end fittings are constructed of polypropylene with 1/4" x 28 threads on one end and a 5/16" hex or square head on the other. The 1/8" end fittings have a 1/8" hole through them to be used with 1/8" TFE tubing. The tubing is to be flared to keep the end fitting and accompanying steel washer from sliding off. These are stocked in several different colors, each with washer.

1/16" Tube End Fitting

Tube end fittings are constructed of polypropylene with 1/4" x 28 threads on one end and a 5/16" hex or square head on the other. The 1/16" end fittings have a 1/16" hole through them to be used with 1/16" TFE tubing. The tubing is to be flared to keep the end fitting and accompanying steel washer from sliding off. These are stocked in several different colors, each with washer.



Coupling

Tube end fittings with an internal $\frac{1}{4}$ " x 28 thread are fitted with nylon coupling.

Indicating Drying Tube

The drying tube on the front panel of the Model 1010 should be replaced or refurbished if one of the following conditions occurs:

- The desiccant material inside has changed color from blue to pink.
- A leak has been found around one of the end fittings of the tube.

To change the drying tube:

1. Pull the tube forward until the tube clamp(s) releases the tube.
2. Disconnect the Luer-Lok fittings between the drying tube and the Teflon tubing routed from the inside of the Model 1010.
3. Discard or replace the old drying tube.
4. Remove the red plugs from the ends of the new drying tube.
5. Reconnect the fittings from the Teflon tubing to the drying tube.
6. Push the drying tube back into the clamp(s).

Drying tubes can be refilled with new desiccant (Drierite®) (10–20 mesh) if desired. To refill tubes:

1. Remove the fitting, the pink seal, and the glass wool from the end of the tube.
2. Empty the old desiccant.
3. Place new desiccant in the tube.
4. Replace the glass wool, the pink seal, and the fitting in the end of the tube.

If the drying tube has developed a leak, depending on the nature of the leak, it may be repaired. This can be determined by a visual inspection of the tube.

Note: A black discoloration at one end of the desiccant in the tube is normal. This discoloration is caused by the reaction of SO_x compounds with the desiccant.



CAUTION:
If the NDIR baseline cannot be adjusted to the desired range, then problems other than zero offset are likely. Refer to NDIR troubleshooting in Chapter 7, "Troubleshooting."



WARNING:
Do not make adjustments with GAIN Potentiometer as this would affect the NDIR detector linearity.



CAUTION:
If NDIR baseline cannot be adjusted to the desired range, then problems other than zero offset are likely. Refer to NDIR troubleshooting in Chapter 7, "Troubleshooting."



WARNING:
Do not make adjustments with GAIN Potentiometer as this would affect the NDIR detector linearity.

NDIR Zero

The NDIR detector zero (baseline) will fluctuate up or down during periods of nonuse. This is due to environmental factors such as operating temperature, how long the NDIR case purge has been on (to expel ambient CO₂), or purity of gases (especially if oxygen is being used in the case of POC analysis). However, under routine operating conditions, the baseline reading should be set between 4,000–8,000 for optimum range and linearity response. This adjustment should be checked after every 100 hours of operation (corresponds to gas service maintenance). To adjust the NDIR baseline:

1. Press [F3] to select **RUN SCREEN**.
2. With the Model 1010 in the standby state, remove the left bay cover to gain access to NDIR detector adjustments.
3. Slowly turn OFFSET adjustment (on IR board) counterclockwise to increase baseline (positive shift) or clockwise to decrease baseline (negative shift) and set output between 4,000–8,000.
4. Allow the Model 1010 to perform several automated analyses and re-check baseline with Model 1010 in the standby state. Make any adjustments if necessary as described above.
5. Replace left bay cover.

Gas Service

Gas consumption is listed in Chapter 1, "Introduction." Standard 2000-psi gas cylinders hold over 200 cubic feet. There are 28.32 liters per cubic foot. Thus, a standard cylinder should last at least 260 hours. Cylinder gas pressure should be monitored after each 100 hours of operation with gas flow to confirm sufficient gas for planned operation.

Sample Pump

This procedure applies to the loop sampling capability which includes a peristaltic pump mounted inside the left bay. It is used to aspirate samples through the loop sampling inlet and the sample loop. The pump housing contains a length of tubing mounted in the housing. The tube is considered expendable because the tubing will eventually wear out.

The tubing should be inspected after every 2000 hours of operation. More frequent inspections may be necessary if running samples containing strong acids or bases.

1. Remove the plastic barb fitting from the end of the outlet leg (closest to outside of left bay) of the black Norprene tube.



CAUTION:
*I/O control board
damage may
occur if the pump
drive assembly
becomes jammed
due to improper
pump tube
installation.*



CAUTION:
*Do not over-
tighten as
damage may
occur to the glass
condenser and
Teflon parts.*

2. With a small, flat-blade screwdriver, carefully pry apart the teeth of the plastic retaining clamp on the inlet leg of the Norprene tube and remove the retaining clamp from the end of the tubing.
3. Press [F7] for the **DIAGNOSTICS SCREEN**.
4. Under the **ACTUATE** section, turn on the **Sample Pump**.
5. While the pump is turning, pull the sample outlet leg to remove the tubing from the pump housing.
6. Inspect the tubing for excessive wear, holes or cracks, and replace if these signs are evident. If the outside of the tube is dry or a replacement tube is being installed, lightly coat the outside wall that will be exposed to the pump housing with a silicone grease lubricant. When installing the new pump tubing, the pump should only be turned on in one or two second increments to allow better control over feeding the pump tube into the housing.

Digestion Vessel and Condensation Chamber

Maintenance of the digestion vessel and condensation chamber assembly should be performed after every 2000 hours of operation to check for signs of degradation.

The correct assembly of the digestion vessel is shown in Figure 5.1. To assemble the digestion vessel after inspection:

1. The short, flared Teflon tubes for directing acid and oxidant into the bottom of the digestion vessel should not touch the sides of the vessel. They should extend down through the digestion vessel cap inside the vessel.
2. The long, flared Teflon tube for sample injection, purging, and draining is to be placed through the side port of the digestion vessel cap. Upon initial installation only, trim this tube to a length such that when the digestion vessel cap and chamber are assembled, the tube just touches the center of the conical bottom with only slight bending of the tube in the digestion chamber.
3. Once the drain tube is cut to its final length, slide the 1/8" x .063" Teflon tubing (Part #147901) onto the drain tube from the bottom.
4. Coil the platinum wire (Part #165581) around the bottom 1/8" (3 mm) of the drain tube.
5. Place the 18-mm nut (Part #224675) and ferrule (Part #224352) on the digestion vessel.
6. Screw the digestion vessel nut onto the digestion vessel cap until the ferrule seals.



7. Insert the inlet of the condensation chamber through the 1/4" stainless steel nut and ferrule into the top of the digestion vessel cap and carefully screw the 1/4" stainless steel nut down into the cap, 1/8-turn past finger-tight.
8. If the 1/4" x 1/8" reducing union was removed from the outlet (top) of the condensation chamber to facilitate the maintenance procedure, tighten 1/8-1/4-turn (max.) past finger-tight. Reconnect the inlet of the permeation tube to the union and tighten 1/4-turn past finger-tight.
9. Confirm proper final position of the drain tube by visual inspection through the digestion chamber.

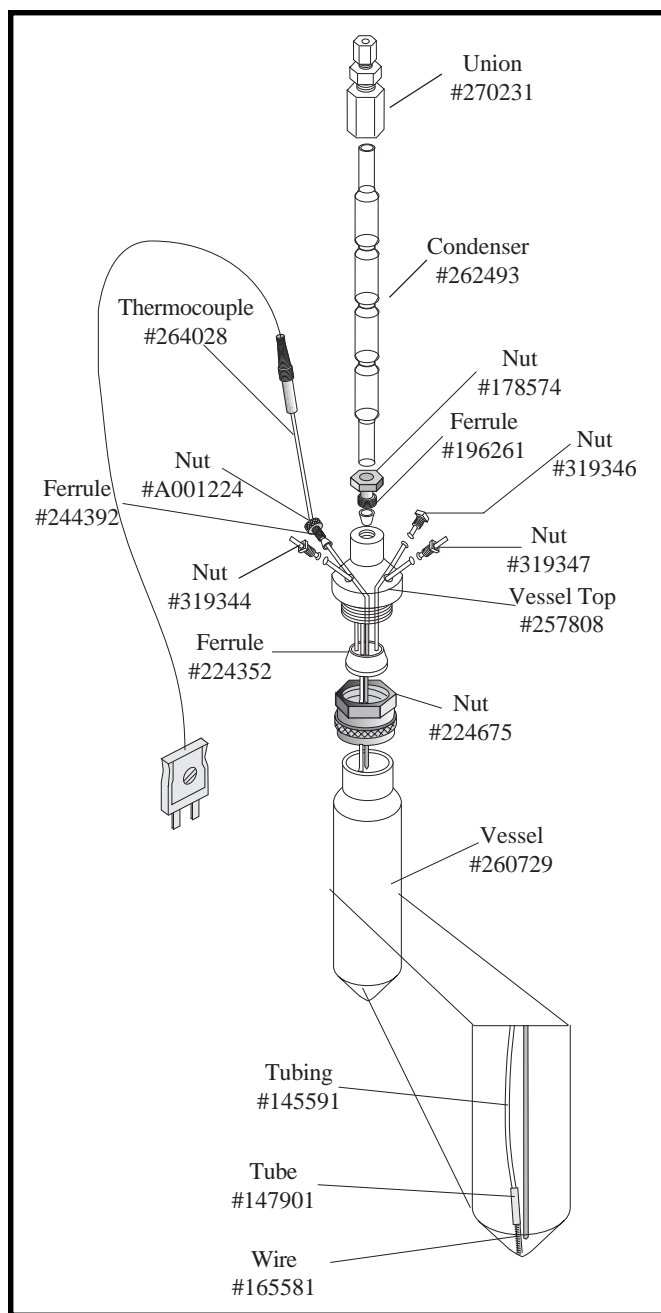


Figure 5.1. Digestion Vessel Assembly

10. Perform a leak test as outlined later in this chapter and correct leaks as necessary.
 11. Apply a thin coating of white silicone heatsink compound to the outside wall of the digestion chamber.
- Note:** It is important to reinstall the digestion vessel with silicone heatsink compound to ensure efficient heat transfer between the heater block and the quartz digestion vessel.
12. Reinstall the digestion vessel/condensation chamber assembly in the heater block.



CAUTION:
Never remove or loosen any part of the tee fittings on the permeation tube except the nuts on each side arm.

Permeation Tube

A gas permeation tube is plumbed between the effluent of the condensation chamber and the indicating drying tube. This is a coaxial tube set containing a hygroscopic membrane in a continuous drying process to selectively remove water vapor from mixed gas streams. The membrane is a proprietary extrudible desiccant in tubular form inside an outer tube shell. When an intermittently wet gas stream flows through the inner tube while a dry gas purges the shell in a countercurrent fashion, water vapor molecules are transferred through the walls of the tubing.

The ion membrane is chemically resistant to all gases and liquids. However, the drying capacity may be decreased if the membrane becomes contaminated with nonvolatile liquids or salts. For this reason, the permeation tube should be cleaned or replaced after every 2000 hours of operation as preventative maintenance.

Inorganic salts that are adsorbed into the membrane can be removed by 10% nitric acid at 50°–60°C as follows:

1. Remove the tube with tee fittings intact from the system.
2. Remove the gas/liquid separator disk (Part #192120) from the barb fitting.
3. Rinse the tube with acid solution and then clean water.
4. Dry the tube under a gas flow.
5. Reinstall the gas/liquid separator disk, pressing the tubing fully onto the barb fitting.
6. Reinstall the tube into the system.
7. Perform a leak check as outlined later in this chapter.

NDIR Detector

Under normal operating conditions, the NDIR detector will not require any scheduled maintenance. If NDIR detector problems are detected or suspected, please contact the OI Analytical Technical Support Department at (800) 336-1911 or (979) 690-1711.

NDIR Linearization Check

All nondispersive infrared analyzers produce a nonlinear response unless electronically corrected by a linearizer board, or in the case of the Model 1010, the output response is corrected algebraically.



The detector in the Model 1010 has been linearized over a range of 0–125 μgC and should remain linearized indefinitely. However, quality assurance practices and proper maintenance procedures should include routine linearity checks. The procedure that follows is recommended.

Note: If a linearity problem is suspected, contact the OI Analytical Customer Service Department for assistance.

1. Using a 5-mL sample loop volume, calibrate the Model 1010 on 10 mL of a 5 ppmC standard (TIC or TOC).

This is mid-scale (50 μgC) of the detector's range, so equivalent standards using appropriate sample volumes can be substituted.

2. When calibration is completed, run 5 mL of the 5 ppmC and 15 mL of the 5 ppmC as a check standard to confirm linearity. If linearity cannot be confirmed, contact the OI Analytical Technical Support Department.

Nonscheduled Maintenance - Mechanical

This section describes procedures for setting and testing certain mechanical components for proper operation if replaced during nonscheduled maintenance (troubleshooting).

Calibrating Reagent Pumps

This calibration applies to both the acid and oxidant pumps. The pumps can be identified by the color of the Teflon lines running to and from the pumps—acid pump lines are red and oxidant pump lines are green.

1. Remove the acid/oxidant line from the top of the digestion vessel ($\frac{1}{8}$ " O.D. line with a red/green fitting).
2. Verify that the acid/oxidant bottle is filled and that the acid/oxidant line has been properly primed.
3. Connect the acid/oxidant fitting to a 1" piece of $\frac{1}{16}$ " O.D. x .030 I.D. Teflon tubing, using a $\frac{1}{4}$ "-28 coupling.
4. Press [F7] for the **DIAGNOSTICS SCREEN**.
5. Under the **PRIME REAGENTS** section, program **Acid** or **Oxidant** for five times.
6. Place the end of the tubing into a measuring vessel and press [ENTER].
7. When the pump stops pumping, measure the volume of the contents in the vessel using a 2-mL syringe.



WARNING:
*Phosphoric acid
and sodium
persulfate are
corrosive
substances;
always wear
appropriate
chemical eye and
skin protection
when handling
these materials.*



8. To adjust the pump volume, loosen the 1/2" locknut on the threaded shaft at the bottom of the pump.
9. If the volume of the vessel is more than 1 mL, turn the 1/4" shaft (with a 1/4" wrench) below the pump clockwise. If the volume of the vessel is less than 1 mL, turn the shaft counterclockwise.
10. Repeat the above steps until the volume dispensed for five strokes is between 0.475 and 0.525 mL.
11. Tighten the locknut and check the volume again.
12. Repeat this procedure for both reagent pumps.

Performing a System Leak Check

1. Press [F7] for the **DIAGNOSTICS SCREEN**.
2. Press [L] for **LEAK CHECK**.
3. Follow the instructions on the screen.

Note: The Model 1010 Start-Up Kit contains a vent plug tube assembly (Part #248864) that is designed to plug the vent port.

If the Model 1010 fails the leak check:

1. Verify that the plug at the vent, labeled (6) in the flow diagram (Figure 5.2), is not leaking.
2. If the plug is not leaking, block the flow at the filter (1).
3. Rerun the leak check.
4. If the leak check fails, the leak is between the gas inlet and the plug. Use the flow diagram and a Snoop[®] to locate the leak.
5. If the leak check passes, remove the block at the filter (1) and block the flow at the sample loop valve (2).
6. Rerun the leak check.
7. If the leak check fails, the leak is between the filter (1) and the plug on the valve. Use the flow diagram and Snoop to locate the leak.
8. If the leak check passes, remove the block at the sample loop valve (2) and block the flow at the digestion vessel (3).
9. Rerun the leak check.

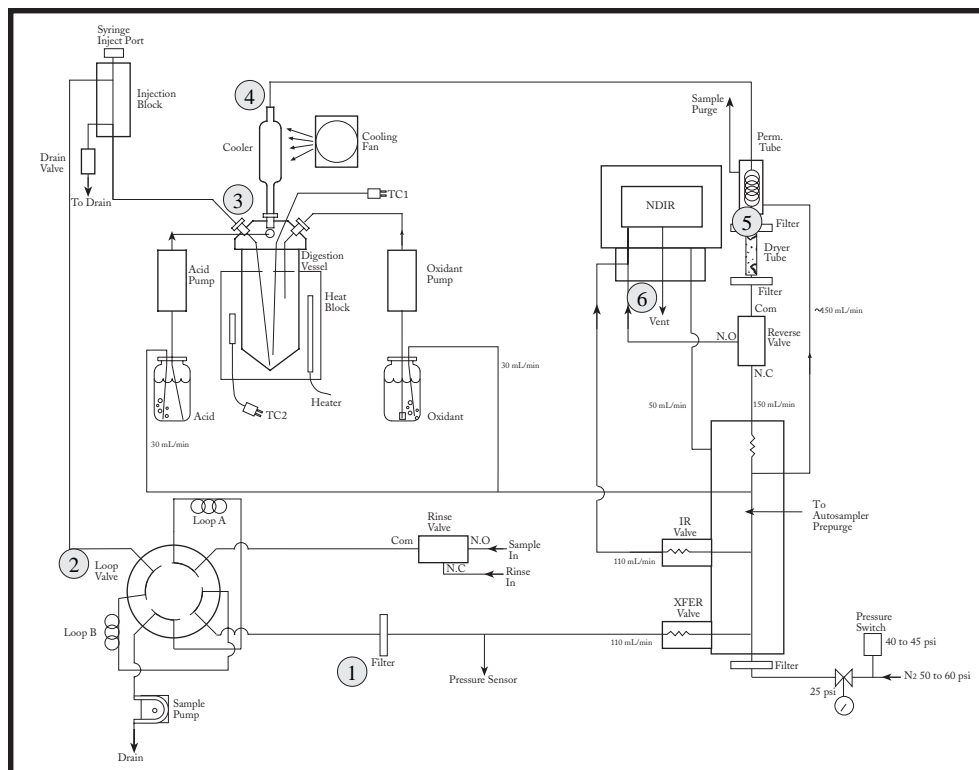


Figure 5.2. Model 1010 Flow Diagram

10. If the leak check fails, the leak is between the valve and the plug on the digestion vessel. Use the flow diagram and Snoop to locate the leak.
11. If the leak check passes, remove the block at the digestion vessel (3) and block the flow at the top of the condensation vessel (4).
12. Rerun the leak check.
13. If the leak check fails, the leak is between the inlet to the digestion vessel (3) and the top of the condensation vessel. Use the flow diagram and Snoop to locate the leak.
14. If the leak check passes, remove the block at the top of the condensation vessel (4) and block the flow between the dryer tube and the permeation tube (5).
15. Rerun the leak check.
16. If the leak check fails, the leak is between the top of the condensation vessel (4) and the permeation tube. Use the flow diagram and Snoop to locate the leak.
17. If the leak check passes, the leak is between the dryer tube and the vent.
18. Remove the block at the dryer tube and use the flow diagram and Snoop to locate the leak.



Changing Sample Loops

The Model 1010 contains an 8-port sample valve with a variety of different loop sizes available. Loop fittings and valve connections are color-coded to prevent incorrect connections.

Converting from 1-mL to 5-mL, 10-mL, or 25-mL Sample Loops

1. Remove the left bay cover from the Model 1010.
2. Locate the two 1-mL sample loops.
3. Locate the fittings (blue and red, respectively) on each loop.
4. Disconnect the blue and red fittings from the $\frac{1}{4}$ -28 fittings in the sample valve.
5. Connect the new loops (5 mL, 10 mL, or 25 mL) by screwing the blue fittings into ports 3 and 7 (loop B) and the red fittings into ports 1 and 5 (loop A) in the 8-port sample valve.
6. Replace the left bay cover.
7. Press [F5] for the configuration screen and change the **Loop Size** to the size of the loop that has been installed.

Flow Adjustment

This procedure is used to verify proper gas flow through the Model 1010 flow paths.

1. Stop any run of the Model 1010 and verify that the unit is in the standby state.
2. Press [F7] for the **DIAGNOSTICS SCREEN**.
3. Under the **ACTUATE** section, turn on the **Transfer Valve** and turn off the **IR (NDIR) Valve**.
4. Verify that the flowmeter now reads 110 ± 1 mL/min. This indicates proper transfer flow.
5. If the transfer flow is not within specification, adjust the main inlet regulator located on the floor of the chassis in the left valve bay of the Model 1010. Adjust until the flow is within range.
6. While still in the **DIAGNOSTICS SCREEN**, turn on the **IR Valve** and verify that all other valves are in the off position.
7. Connect a flowmeter to the fitting labeled “VENT” on the front panel of the Model 1010.



8. Verify that the flowmeter reads 110 ± 10 mL/min. This indicates proper NDIR flow. If not within specification, there may be a problem with the frit in the manifold. Contact the OI Analytical Customer Service Department for assistance.
9. Measure the reagent purge flow by connecting the flowmeter to the end of the reagent purge line ($\frac{1}{16}$ "-line in reagent bottle). The flow should be in the range of 50 ± 20 mL/min. Repeat the procedure for each reagent.
10. Connect the flowmeter to the "PURGE OUT" line located on the front panel of the Model 1010.
11. The flowmeter should read 200 ± 25 mL/min. If not, check for a tubing restriction. (This is the dry purge flow through the permeation tube.)
12. Connect the flowmeter to the sample drain (reverse valve) output line located on the Model 1010 back panel.
13. While still in the **DIAGNOSTICS SCREEN**, turn on the **Drain Valve** and the **Reverse Valve**.
14. The flowmeter should read 170–200 mL/min. If not within specification, check the frit restriction in the manifold. Contact the OI Analytical Technical Support Department for assistance.

Calibrating Sample Loops (1 mL, 5 mL, 10 mL, or 25 mL)

Sample loops, other than the loops that came with the unit, installed on the Model 1010 must be calibrated on the Model 1010 to ensure accurate sample volume.

1. Press [F5] for the **CONFIGURATION SCREEN**.
2. Under the **SAMPLE INTRODUCTION** section, select **Sipper**.
3. Press [F7] for the **DIAGNOSTICS SCREEN**.
4. Press [C] for **CALIBRATE LOOP**.
5. Follow the instructions on the screen.

Note: The loop calibration routine for 1-mL and 5-mL loops on the Model 1010 fills the loop five times. Divide the liquid measurement by five for the actual volume. The 10-mL sample loops are filled twice (divide the liquid measured by two). The 25-mL loops are filled only once (no division is required).



Note: The sample loops must be matched as follows:

Sample Loop Volume (mL)	Loop Volume Specification (mL)	Difference Between Loops (mL)
1.00	±0.05	±0.010
5.00	±0.10	±0.020
10.00	±0.20	±0.040
25.00	±0.50	±0.125

6. Once the specification for the loops is met, enter the **Loop** volumes, under the **SAMPLE LOOPS** section on the diagnostics screen.

Restrictions in the System

If the Model 1010 detects a restriction in the gas pathway, a warning screen will appear. This warning will appear only if the system pressure exceeds 20 psi. If this warning does appear, the restriction must be corrected before resuming operation. To locate the restriction:

1. Press [F7] for the **DIAGNOSTICS SCREEN**.
2. Under the **CURRENT READING** section, view the **Pressure** display.
3. Use the flow diagram and disconnect tubing in the places described under “Performing a System Leak Check,” beginning at the vent.
4. Isolate the restriction and remove it.
5. Gas pressure should return to below 1.0 psi.

Nonscheduled Maintenance - Electronic ██████████

This section describes procedures for testing and calibrating electronic components for proper operation if replaced during nonscheduled maintenance (troubleshooting).

Maintenance requirements of the Model 1010 are not extensive. Visual inspections mainly include the suggested maintenance procedures. Electronic realignment is not generally required on a regular schedule and need not be performed unless certain circuit boards or components are replaced, or the Model 1010 shows signs of misalignment in the analog circuitry.

Printer Checkout

The printer supplied with the printer option has a programmed self-test routine. It is activated as outlined in the printer manual.



Chapter 7

Troubleshooting

This section contains a list of symptoms, and their most probable causes and corrections. Before using this section, the operator should become thoroughly familiar with the operation and maintenance information contained in previous chapters.

System Performance Symptoms

Symptom	Probable Cause	Corrective Action
No response	No gas flow Wrong reagents being used	Check gas source See specific components later in this chapter. See Chapter 1, "Introduction"
Nonreproducible response for both TIC and TOC	Sample volume not constant Leak in system Insufficient acid to completely liberate CO ₂ Insufficient react time for high TIC samples Insufficient react time for complete TOC oxidation	Improve syringe technique Check correct loop size selection Check sample loop for complete filling when sample pump is on Inspect pump cartridge for leaks or wear. See "Sample Pump Housing Maintenance" in Chapter 6 Perform leak check Increase acid volume. Check acid volume by performing "Calibration of Reagent Pumps" procedure in Chapter 6 Extend react times Extend react time



Symptom	Probable Cause	Corrective Action
<p>Nonreproducible response for both TIC and TOC (cont.)</p>	<p>Oxidant from previous sample not completely drained</p> <p>Contaminated digestion vessel</p>	<p>Reposition tube inside digestion vessel. See “Digestion Vessel Maintenance” in Chapter 6</p> <p>Allow Model 1010 to perform clean water cycling as described in Chapter 5, “Operation”</p>
<p>Nonlinear response for TIC and TOC</p>	<p>NDIR baseline set too high</p> <p>Blanks not properly determined or entered</p> <p>Carbon mass exceeds linear range of detector</p> <p>NDIR not properly linearized</p>	<p>Adjust NDIR zero to 4,000–8,000 in standby condition. See Chapter 6, “Maintenance”</p> <p>Run blanks</p> <p>Refer to tables in Chapter 5, “Operation,” for selection of sample volume. Reduce sample size or dilute sample</p> <p>Confirm linearity following NDIR Linearization. See Chapter 6, “Maintenance”</p>
<p>Negative values display or printed</p> <p>Low response for both TIC and TOC</p>	<p>Improper blank value in memory (too high)</p> <p>Inaccurate high blank values for TIC and TOC</p> <p>Inaccurate low calibration</p> <p>Inaccurate high sample volume entered</p>	<p>Run blanks</p> <p>Run blanks</p> <p>See Chapter 5, “Operation,” for calibration procedure</p> <p>Enter proper sample volume. See Chapter 5, “Operation”</p>



Symptom	Probable Cause	Corrective Action
<p>Low response for TIC with normal or reproducible response for TOC</p>	<p>Restriction in sample gas lines</p> <p>Insufficient acid addition to completely liberate CO₂</p> <p>Insufficient react time</p> <p>Improper acid reagent</p> <p>Faulty acid pump</p> <p>TIC mass exceeds linear range detector</p>	<p>Remove restriction in system. See “Restrictions in the System” in Chapter 6</p> <p>Increase acid volume</p> <p>Extend react time</p> <p>Confirm that 5% (vol/vol) phosphoric acid is being used</p> <p>Check acid pump calibration. See Chapter 6, “Maintenance”</p> <p>Refer to tables in Chapter 5, “Operation,” for selection of sample size. Reduce sample size or dilute sample</p>
<p>Low response for TIC with high response for TOC</p>	<p>Insufficient acid to completely liberate CO₂</p> <p>Insufficient react time for high TIC samples</p> <p>Faulty acid pump</p>	<p>Increase acid volume</p> <p>Extend purge time</p> <p>Check acid pump calibration. See Chapter 6, “Maintenance”</p>
<p>Low response for TOC with normal response for TIC</p>	<p>Incomplete oxidation, not enough oxidant</p> <p>No persulfate reagent or improper reagent in oxidant reagent bottle</p> <p>Incomplete oxidation, insufficient reaction time</p> <p>Faulty oxidant pump</p>	<p>Increase oxidant volume</p> <p>Confirm that correct persulfate solution is in oxidant reagent bottle. See Chapter 1, “Introduction,” for reagent and materials required</p> <p>Extend react time</p> <p>Check oxidant pump calibration. See Chapter 6, “Maintenance”</p>



Symptom	Probable Cause	Corrective Action
Low response for TOC with normal response for TIC (cont)	TOC mass exceeds linear range of detector Digestion vessel not heating	Refer to tables in Chapter 5, "Operation," for selection of sample size. Reduce sample size or dilute sample Check sample temperature
Low TOC response with high TIC response	Oxidant from previous sample not completely drained Reagent bottles switched	Reposition tube inside digestion vessel. See "Digestion Vessel Maintenance" in Chapter 6. Check purge gas flow during draining, note any excessive restriction preventing good draining of vessel Confirm acid and persulfate in correct bottle to respective pumps
High response for TIC and TOC	Wrong low blank values for TIC and TOC Wrong high response factor Wrong low loop size entered System contamination	Run blanks Recalibrate Model 1010 Enter proper loop size Perform visual inspection of all surfaces which contact sample and clean as needed with hot water Perform clean water cycling routine described in Chapter 5, "Operation"
High TIC blanks	CO ₂ in acid CO ₂ in purge gas	Confirm reagent bottle purging. Purge CO ₂ from acid Install ascarite scrubber in-line or use higher quality gas



Symptom	Probable Cause	Corrective Action
High TOC blanks	<p>CO₂ in oxidant</p> <p>CO₂ in purge gas</p> <p>Organic carbon in oxidant</p> <p>Organic carbon in acid</p> <p>Digestion vessel contaminated</p> <p>Drain line in digestion vessel not positioned properly (i.e., carryover)</p>	<p>Confirm oxidant bottle is being purged. Purge CO₂ from reagent</p> <p>Install ascarite scrubber in-line or use higher quality gas</p> <p>Clean oxidant of organics. See “Reagents and Materials” in Chapter 1</p> <p>Clean acid of organics. See “Reagents and Materials” in Chapter 1</p> <p>Cycle analysis mode with extended digestion time. See “Clean Water Cycling” in Chapter 5 to clean vessel</p> <p>See “Digestion Vessel Maintenance” in Chapter 6</p>
Wrong values for TC only analysis	When system is in TC mode, all possible TIC/TOC blank problems must be considered	When in TC mode, test solutions for high TIC blanks as well as for high TOC blanks
Model 1010 will not power up	<p>Model 1010 not plugged into appropriate line voltage</p> <p>Blown fuse</p>	<p>Check power cord connection</p> <p>Check power breaker to plug outlet. Reset if tripped</p> <p>Check A/C power control board fuses and replace if blown. See Chapter 2, “Description of Components,” for location</p>



System Component Symptoms

Symptom	Probable Cause	Corrective Action
Sample does not properly aspirate into Model 1010 from sample bottle or autosampler	Incorrect sample loop size entered	Enter correct values
	Worn sample pump tubing	Replace sample pump tubing as described in “Sample Pump Tubing Maintenance” in Chapter 6
	Leak in sampling line	Leak-check sampling tubing from loop injection port to sample pump
	Sample loop not properly tightened	Check sample loop connections for finger-tightness
	Pump head tubing pinched shut from lack of use	Service or replace pump tubing. See Chapter 6, “Maintenance”
“W” shaped or negative inflection on CO ₂ peak (TIC, TOC, or POC)	Gross CO ₂ contamination in gas	Change gas cylinder. Use gas with 99.98% + purity
Water dripping from injection port	Leaky septum	Replace septum as outlined in “Changing Injection Port Septum” in Chapter 6
NDIR baseline zero too high (greater than 8,000 with no adjustment)	Contaminated NDIR sample cell	Contact OI Analytical Technical Support Dept
	Digestion vessel heater too hot	Check sample temperature. It should be 92°–99°C in TOC react
	Digestion vessel drain tube not positioned properly - insufficient draining of vessel	See “Digestion Vessel Maintenance” in Chapter 6



Warning Screens

Warning Screen Using Keyboard/Monitor	Warning Screen Using WinTOC	Problem/Solution
Warning: Heating Circuit Overtemp	Warning: Sample heater block over max temp.	The digestion vessel heater has overheated beyond its set point. Contact the OI Analytical Customer Service Department.
Warning: Low Gas Pressure	Warning: Low gas pressure.	The gas supply has dropped below the required pressure to operate the Model 1010. If the Model 1010 is running an analysis, it has subsequently stopped the analysis and drained the digestion vessel. Replenish gas supply.
Warning: Printer Error	Warning: Attached 1010 printer error.	The printer is not able to print data. The Model 1010 will store up to ten pages of data in this mode. After the ten pages, the Model 1010 will stop any analysis and return to the standby state.
Warning: IR (NDIR) Failure: MAX OUTPUT	Warning: Possible IR detector failure.	The NDIR output has reached maximum output (65535) and remains at that point. Contact OI Analytical Technical Support Department.
Warning: IR Failure: NO OUTPUT	Warning: Possible IR detector failure.	The NDIR output has reached zero (0) and remains at that point. Contact OI Analytical Technical Support Department.



Warning Screen Using Keyboard/Monitor	Warning Screen Using WinTOC	Problem/Solution
Warning: Possible Gas Restriction	Warning: Possible gas flow restriction.	The Model 1010 has detected a restriction in the system that could possibly cause a problem with the operation of the unit. See “Restrictions in the System” in Chapter 6.
Warning: Reported Mass Over 130 µgC	Warning: The analysis has exceeded the linear range (>130 µgC) of the analyzer.	The Model 1010 has detected a mass of carbon out of its linear range (125 µgC). The concentration and mass of the sample causing this display may not be accurate. See Chapter 5, “Operation,” for selecting the correct sample volume.
Warning: No Sequence Loaded	Not applicable	The Model 1010 has no sequence loaded. See Chapter 5, “Operation,” for programming a run.
Warning: Loop Size Has Changed	Not applicable	The operator has changed the loop size on the configuration screen. Other parameters, such as loop volume, rinse volume, and standard volumes may need to be adjusted.
Warning: Tray changed...Recalibrate	Warning: Autosampler is not calibrated.	The “Home” position on the Model 1051 Autosampler carousel needs to be calibrated.
Warning: IR Baseline Too High	Warning: The IR signal is currently above the maximum allowed baseline (10,000).	The NDIR output is too high (greater than 10,000); as such a sequence cannot be started before the IR baseline value settles down.



Chapter 8

Replacement Parts

This chapter lists the order numbers for replacement parts and support items for the Model 1010 Wet Oxidation TOC Analyzer and its associated options. Replacement parts considered as expendable (XPND) are marked with an asterisk. (Expendable components should be replaced regularly or are easily broken or deformed.) A supply of XPND parts should be kept on hand. Units of measure (U/M) are also given.

Replacement Parts

<u>Part Name</u>	<u>Part #</u>	<u>U/M</u>	<u>XPND</u>
Boards			
Infrared Detector Assembly	286922	ea	
Interface	265477	ea	
PC104 Controller Assembly	306217	ea	
Parallel Interface	296327	ea	
RAM Memory	289538	ea	
Digestion Vessel Assembly			
Assembly - Digestion Vessel	262105	ea	
Clamp - Condenser Mounting	180604	ea	
Condenser - Digestion Vessel	262493	ea	*
Ferrule - 1/4" Teflon Tube	196261	5/pk	*
Ferrule - 18 mm Teflon Tube	224352	10/pk	*
Ferrule - Tefzel 0.084	244392	ea	*
Nut - Green Tube End Fitting 1/8"	166357	ea	*
Nut - Red Tube End Fitting 1/8"	166365	ea	*
Nut - Red Tube End Fitting 1/16"	166307	ea	*
Nut - 1/4" Stainless Steel	178574	ea	
Nut - 18 mm Stainless Steel	224675	ea	
Nut - 1/4"-28 x 0.100 PEEK	A001224	ea	
Thermocouple - Digestion Vessel	264028	ea	
Tube - (Drain Line Support) Teflon	147901	ft	*
Tubing - Teflon, 1/16 x .031 I.D.	145591	ft	*
Union - 1/4"-1/8" Polypropylene	270231	ea	
Vessel - Digestion Vessel	260729	ea	*
Vessel Top - Digestion Vessel	257808	ea	*
Wire - Platinum Wire, 0.008" Diameter	165581	ft	



<u>Part Name</u>	<u>Part #</u>	<u>U/M</u>	<u>XPND</u>
Electronics			
Block - Heater Block	260703	ea	
Drive - Floppy Drive (3.5")	266080	ea	
Fan Assembly - Radial Cooling Fan	262147	ea	
Heater - Cartridge Heater for Heater Block	263046	ea	
Keyboard - 83-Key	273557	ea	
Monitor - 9" Monochrome	273540	ea	
Power Cord - North America Type	116038	ea	
Power Supply - 110 W	305131	ea	
Power Supply - 25 W, 15 V (PCA)	301663	ea	
Relay - Heater Block	272138	ea	
Thermocouple - Type K 24"	262980	ea	*
Ferrules			
1/16" Swagelok Back, Brass	196162	5/pk	
1/16" Swagelok Front, Brass	196170	5/pk	
1/8" Swagelok, Brass	196089	10/pk	*
1/8" Swagelok, Stainless Steel	210591	5/pk	*
1/4" Teflon Tube	196261	5/pk	*
1/8" Tefzel	317545	ea	*
18 mm Teflon Tube	224352	10/pk	*
Tefzel 0.084	244392	ea	*
Fittings - Adapters			
1/16 Barb, Kynar, Luer, Female	196386	ea	*
1/16 Barb, Kynar, Luer, Male	194415	ea	*
1/16 Bulkhead, Kynar, Luer	197798	ea	
1/16 to (2)1/16, Kynar	270496	ea	*
1/8 MNPT-F10-32, Brass, Male/Female	166208	ea	*
1/8 Tube-32, Brass/Nickel	196600	ea	
10-32 x 1/16 Hose, Brass	166191	ea	
Fittings			
Coupling 1/4-28, Polypropylene	166274	ea	*
Injection Port 1/4-28, 22 Gauge, Kel-F	270777	ea	*
Nut 1/4"-28 x 0.100 PEEK	A001224	ea	
Nut 1/8 Female, Brass	128108	ea	
Nut 1/16 Male, Brass/Nickel	196303	5/pk	
Nut 1/8, Polypropylene	274613	ea	
Nut 1/4, Stainless Steel	178574	ea	
Nut 18 mm Female, Stainless Steel	224675	ea	
Plug 1/4-28, Tefzel	166430	ea	*
Port 1/8, Brass	117721	ea	
Tee 1/8 Tube Male, Brass	124750	ea	
Tee NDIR, Kel-F	260885	ea	*
Union 1/4-1/8 Tube Male, Stainless Steel	124735	ea	
Union 1/8 Hose, Bulkhead, Brass/Nickel	225565	ea	
Union 1/8-1/16, Stainless Steel	178178	ea	
Union 1/16 Bulkhead, Brass	175803	ea	
Union 1/8, Brass	124420	ea	



<u>Part Name</u>	<u>Part #</u>	<u>U/M</u>	<u>XPND</u>
Fittings - Tube End Nuts			
Nut PEEK 1/8" Red	319344	ea	
Nut PEEK 1/8" Green	319347	ea	
Nut PEEK 1/8" Yellow	319346	ea	
Nut PEEK 1/8" Natural	319343	ea	
Nut PEEK 1/8" Blue	319345	ea	
Tubing			
Tubing TFE 1/8 x 0.0632Green	319606	ft	
Tubing TFE 1/8 x 0.062 Yellow	319329	ft	
Tubing TFE 1/8 x 0.062 Blue	319607	ft	
Tubing TFE 1/8 x 0.045 Clear	319851	ft	
Tubing TFE 1/8 x 0.062 Black	319293	ft	
Tubing TFE 1/8 x 0.062 Red	319328	ft	
Software/Firmware			
Model 1010 Firmware	250837	ea	
Model 1010 Firmware Upgrade	280941	ea	
WinTOC Software for Model 1010	250613	ea	
WinTOC Software Upgrade	280933	ea	

Supplies and Options

Analyzer Kits

Flaring Tip 1/16"	169137	ea	*
Flaring Tip 1/8"	169129	ea	*
Flaring Tool, Heats 1/16" and 1/8" Flaring Tips	168808	ea	
Gas Cylinder Regulator, Helium/Nitrogen,	144585	ea	
SS Diaphragm, 1/8" Tube Fitting	319857		
Gas Cylinder Regulator, Oxygen, SS	150326	ea	
Kit, Install	250605	ea	
Kit, Reagent Pump Rebuild	178806	ea	

Autosampler Supplies - 14-mL Vials

Caps - Open-Hole Screw	174558	100/pk	*
Septa - Teflon-Faced (0.065 mm)	258574	100/pk	*
Vials	210070	250/box	*

Autosampler Supplies - 40-mL Vials

Caps - Open-Hole Screw	296079	72/pk	*
Septa - Teflon-Faced (0.065 mm)	258566	100/pk	*
Septa - Teflon-Faced (0.065 mm)	173211	50/pk	*
Vials - VOA Autosampler Vials	296087	72/box	*

Autosampler Supplies - 100-mL Vials

Caps - Open-Hole (Crimp)	295857	1,000/pk	*
Septa - Teflon-Faced	295865	100/pk	*
Vials	295840	1,000/box	*



<u>Part Name</u>	<u>Part #</u>	<u>U/M</u>	<u>XPND</u>
Chemicals and Reagents			
Ascarite - For Gas Filter	110122	500 g	
Phosphoric Acid, 85%	110080	500 mL	
Phosphoric Acid Solution - 5%, Cleaned	169244	1 L	
Potassium Biphthalate (KHP) - 1,000 ppmC	169252	10 mL	
Potassium Biphthalate (KHP) Crystals	136954	500 g	
Sodium Carbonate - 1000 ppmC	169294	10 mL	
Sodium Carbonate, Anhydrous	136962	500 g	
Sodium Persulfate, Crystals	178848	500 g	
Sodium Persulfate Solution - 100 g/L, Cleaned	169236	1 L	
Halide Scrubber Option Parts			
Cap - Vial Cap	263244	ea	*
Clip - Mounting	271486	ea	
Nut - Polypropylene, 1/4-28 1/8" Tube Clear	165862	ea	
O-ring - Silicone 3/4" x 3/32"	177297	ea	
Shot - Copper, 2-4 mm, 100 g	265223	ea	*
Sparger - 25 mL	263228	ea	*
Infrared Detector Assembly			
Assembly - Complete	286922	ea	
Chamber - 25 mm O.D. x 17.5 mm	262048	ea	
Fitting - Adapter, 10-32 x 1/16" Hose, Brass	166191	ea	
O-ring - Sealing	116400	ea	*
Window - IR Cell 20 mm	173287	ea	*
Miscellaneous Parts			
Check Valve/Filter Kit - Reagent Pump	182253	ea	
Clamp Drying Tube	180604	ea	
Reagent Bottle Caps	265157	ea	
Scrubber Tube, Ascarite	169145	ea	*
Snoop, 8 oz Squirt Bottle	129767	ea	*
O-rings			
Buna-N - Infrared Detector	116400	ea	*
Viton - Manifold 0.042 x 0.042	236604	ea	*
Viton - Manifold 0.156 x 0.070	288068	5/pk	*
Viton - Reagent Bottle Reservoir	210310	5/pk	*
Viton - Reagent Pump	210286	5/pk	*
Viton - Reagent Pump Seal	210302	5/pk	*
Viton - Reagent Pump Rebuild Kit	178806	ea	
Plumbing			
Acid/Oxidant Reagent Bottle Assembly	262030	ea	*
Acid/Oxidant Reagent Metering Pump	182550	ea	
Check Valve - Polypropylene	182238	ea	
Drying Tube - Indicating	273417	ea	*
Filter - In-Line 6A	242941	ea	
Filter - In-Line 10-µ Reagent Bottle	182246	ea	*



Part Name	Part #	U/M	XPND
Filter - Solvent, 7 μ	165656	ea	*
Filter - Teflon In-Line	192120	ea	*
Injector - Block Assembly	263384	ea	*
Injector - Block	260919	ea	*
Permeation Tube Assembly	264119	ea	*
Sample Pump	263442	ea	
Septum - Teflon-faced Silicone, 5 mm x 0.125"	174566	50/pk	*

Printer Supplies/Options

Interface Cable - Parallel/Centronics Type	273227	ea	
Paper - 9 $\frac{1}{2}$ " x 11"	138546	400 sheets	*
Paper - 9 $\frac{1}{2}$ " x 11"	138554	2500 sheets	*
Ribbon Cartridge - Dot Matrix	178871	ea	*

Syringes

10 μ L - 2" Needle	167545	ea	*
25 μ L - 2" Needle	110205	ea	*
50 μ L - 2" Needle	110171	ea	*
100 μ L - 2" Needle	110221	ea	*
500 μ L - 2" Needle	137069	ea	*
2.5 mL - 2" Needle	137051	ea	*

Tubing and Tube Assemblies

Pump Tubing - Norprene	277319	ea	*
Sample Loops - 1 mL w/Ferrule, 1 pair	319737	ea	*
Sample Loops - 5 mL w/Ferrule, 1 pair	319736	ea	*
Sample Loops - 10 mL w/Ferrule, 1 pair	319739	ea	*
Sample Loops - 25 mL w/Ferrule, 1 pair	319740	ea	*
Tube Assembly - Ascarite Scrubber	169145	ea	*
Tube Assembly - Permeation	264119	ea	*
Tube - Norprene [®] Sample Pump $\frac{1}{4}$ " I.D.	277319	ea	*
Tubing - Polypropylene, $\frac{1}{8}$ x $\frac{3}{32}$ Clear	189738	ft	*
Tubing - Teflon, $\frac{1}{16}$ x 0.009 I.D.	303057	ft	*
Tubing - Teflon, $\frac{1}{8}$ x 0.063 I.D.	147901	ft	*
Tubing - Tygon, $\frac{1}{4}$ x $\frac{1}{8}$ I.D.	257469	ft	*

Valves and Valve Assemblies

Assembly - 8-Port, DC Actuator	319735	ea	
Assembly - Manifold Valving	270215	ea	
Assembly - Solenoid 3-Way Pre-Purge	263129	ea	
(Needle Wash, Reverse, Rinse)			
Clip - Mounting Clip, Drain Valve	271486	ea	
Valve - Br/Ni 2-way Manifold (IR and Transfer)	315234	ea	
Valve - Drain Valve	263111	ea	
Valve - 8-Port Valve	319733	ea	
Valve - 2-Way Solenoid (Septum Valve)	319620	ea	
Valve - Polypropylene Check Valve	182238	ea	
Valve Assembly - 8 port, 5 mL with Ferrule	319738	ea	



Notes



Index

A

Alarm Relay Contacts 3
Alarm Relay Option, Installing 22
Analytical Hints 51
Analyzing Reagent Blanks 41
Applications 3
Attenuation 25
Autosampler, Installing 22
Autosampler Interface Connector 17

B

Back View - Model 1010 17
basic samples 53
Bootup Screen 24

C

Calibration 42
 Constant Volume Procedure 46
 TIC Method 46
 TOC Method 46
Calibration Results Screen 34
Calibration Sequence, Running 47
Check Standards, Running 48
Clean Water Cycling 51
Communications 4
Compliance 5
Components, Description of 15
Condensation Chamber Maintenance 60
Condensation Vessel 15
Configuration Screen 29

D

Definitions 8
Description of Components 15
Diagnostics Screen 35
Difficult Samples 52
Difficult Samples, Confirming Recoveries in 51
Digestion Vessel 15
Digestion Vessel Cover 15
Digestion Vessel Heater Block 16
Digestion Vessel Maintenance 60
Dimensions 3



Dissolved Organic Carbon 8
DOC. *See Dissolved Organic Carbon*
Drain 39
Drying Tube 16
Drying Tube Maintenance 58

F

Features 1
 WinTOC 3
Firmware 23
Firmware Screens 23
Fluid Connections 21
Front View - Model 1010 15
Fuse Holder 17

G

Gas Connections 21
Gas Inlet 17
Gas Maintenance 59
Gas Saver 36

I

Injection Port Septum Maintenance 56
Installation 19
 Alarm Relay Option 22
 Autosampler 22
 External Options 22
 Keyboard 22
 Materials Needed Before 20
 Monitor 22
 Printer 22
Instrument Covers, Removing 19
Interferences 11
 Calibration 11
 Method 11

K

Keyboard Cable Interface 18
Keyboard, Installing 22



M

- Maintenance 55
 - Nonscheduled - Electronic 68
 - Nonscheduled - Mechanical 63
 - Calibrating Reagent Pumps 63
 - Calibrating Sample Loops 67
 - Changing Sample Loops 66
 - Cleaning the NDIR Detector Sample Cell 68
 - Flow Adjustment 66
 - Performing a System Leak Check 64
 - Restrictions in the System 68
 - Routine Maintenance 55, 60
 - Condensation Chamber 60
 - Drying Tube 58
 - Injection Port Septum 56
 - NDIR Cell 62
 - NDIR Linearization Check 62
 - NDIR Zero 59
 - Permeation Tube 62
 - Reagent Reservoir 56
 - Sample Pump 59
 - Tube End Fittings 56
- Method 9, 50
- Monitor, Installing 22
- Monitor Interface Connector 18
- Monitor Power Outlet 18

N

- NDIR. *See Nondispersive Infrared Detector*
- NDIR Cell Maintenance 62
- NDIR Linearization Check 62
- NDIR Zero 59
- Nondispersive Infrared Detector 10
- Nonpurgeable Organic Carbon: 9
- NPOC. *See Nonpurgeable Organic Carbon:*

O

- Operation 37
- Options 5

P

- Performance Specifications 4
- Permeation Tube Maintenance 62
- POC. *See Purgeable Organic Carbon*
- Pre-Acid Outlet 16
- Precision 4
- Principle of Operation 1



Printer Checkout 68
Printer, Installing 22
Printer Interface Connector 18
Printer Specifications 5
Pump Drain Line 18
Purge Line Outlet 16
Purgeable Organic Carbon 9

R

Range 4
Reagent Blank 9
Reagent Inlet Line 18
Reagent Pumps, Calibrating 63
Reagent Purge Outlet 18
Reagent Reservoir Maintenance 56
Reagents 11
Replacement Parts 77
Requirements 4
 Gas 4
 Power 5
Rinse 39
Rinse Line Inlet 16
Rinses Per Replicate 54
Rinses Per Sample 54
Run Screen 25

S

Safety Information 5
 Precautions
 Chemical 7
 Compressed Gas Cylinders 7
 General 6
 Operator 6
Safety Symbols 8
Sample Drain Line 18
Sample Inlet 16
Sample Introduction 4, 37
Sample Loops, Calibrating 67
Sample Loops, Changing 66
Sample Pump Maintenance 59
Sample Size 4
Samples containing long chain or complex carbons 53
Samples, Running 49
Samples with pH problems 53
Screen
 Bootup 24
 Calibration Results 34
 Configuration 29
 Diagnostics 35
 Run 25



Sequence 27
Sequence 51
Sequence Screen 27
Sequencing 49
SOC. *See Suspended Organic Carbon*
Specifications 3
Standard 9
Standard Additions 54
Standard, Programming 47
Standards 12
Starting Up 41
Startup Screen 24
STATUS 25
Suspended Organic Carbon 9
Syringe Injection Port 16
System Leak Check 64

T

TC. *See Total Carbon*
TC Detect 30, 39
TC React 30, 39
TIC. *See Total Inorganic Carbon*
TIC Detect 30, 38
TIC React 30, 38
TOC. *See Total Organic Carbon*
TOC Detect 30, 38
TOC React 30, 38
Total Carbon 8, 10
Total Inorganic Carbon 8, 9
Total Organic Carbon 8, 9
Troubleshooting 69
 System Component Symptoms 74
 System Performance Symptoms 69
 Warning Screens 75
Tube End Fittings Maintenance 56

U

United States Pharmacopeia (USP) Standards 12

V

Vent Flow Outlet 16

W

Warning Screens 75
Water Blank 9
Weight 3



P.O. Box 9010
College Station, TX 77842-9010
Tel: (979) 690-1711 • FAX: (979) 690-0440 • www.oico.com