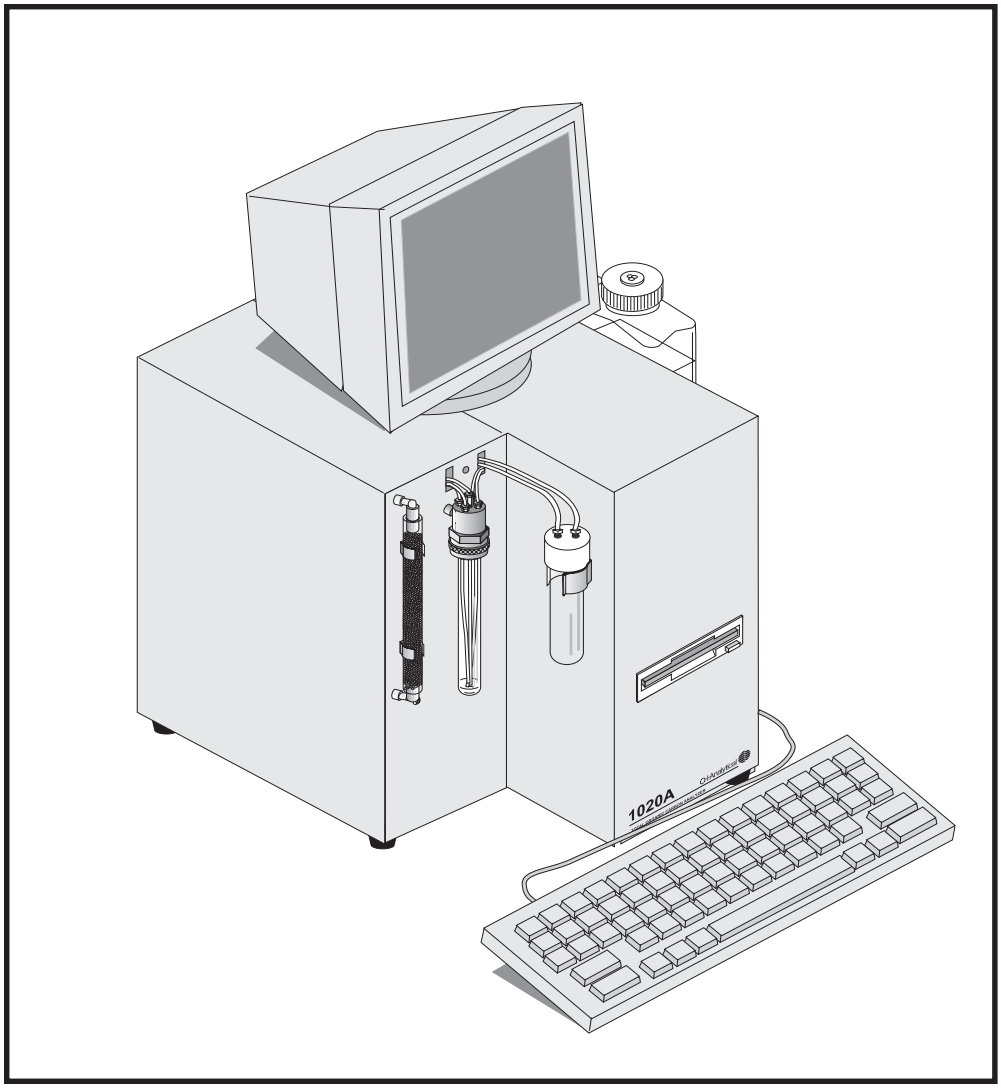
Model 1020A Combustion Total Organic Carbon Analyzer Operator's Manual



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OI Analytical warrants each Model 1020A Combustion 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 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.

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Chapter 1
Introduction

The OI Analytical Model 1020A Total Organic Carbon Analyzer is an automated system for the selective analysis of total carbon (TC), total organic carbon (TOC), total inorganic carbon (TIC), and purgeable organic carbon (POC) in aqueous samples. The Model 1020A TOC Analyzer employs high temperature combustion oxidation, coupled with nondispersive infrared detection technology, for efficient, selective detection of carbon in a wide variety of matrices. This combustion instrument was designed specifically to comply with USEPA, Standard Methods, and ASTM methodologies for the analysis of water with higher levels of carbon as found in wastewater and marine waters.

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 1020A using the keyboard and monitor.

Principle of Operation

Fundamentally, all TOC analyzers perform two tasks: oxidation of organic carbon to form carbon dioxide and the subsequent detection of the carbon dioxide. The methods of oxidation and detection, however, vary greatly and are somewhat application specific.

The OI Analytical Model 1020A TOC Analyzer is a combustion system in which an aqueous sample is introduced to a furnace that is heated to 680°C in an oxygen-rich environment. Water is evaporated and removed from the system, and the total carbon content of the aliquot is oxidized to form carbon dioxide. The carbon dioxide is then swept quantitatively to the infrared cell and is selectively detected. The time-integrated absorbance of the carbon dioxide flowing through the nondispersive infrared (NDIR) detector is directly proportional to the amount of carbon in the sample.

Inorganic carbon exists in solution as carbon dioxide, carbonate, and bicarbonate ions according to Equation 1.1.

EQUATION 1.1

$$CO_2 \leftrightarrow HCO_3^- \leftrightarrow CO_3^{2-}$$

Quantifying inorganic carbon requires shifting the aqueous equilibrium to the left through acidification of the sample, followed by purging and transfer of the carbon dioxide to the NDIR detector.

TOC is determined by the difference between separate TC and TIC analyses. This results in a true TOC value, inclusive of NPOC.

The NDIR detector is a highly selective Golay cell composed of an infrared source, chopper, absorption flow cell, and sensor assembly. (See Figure 1.1.)

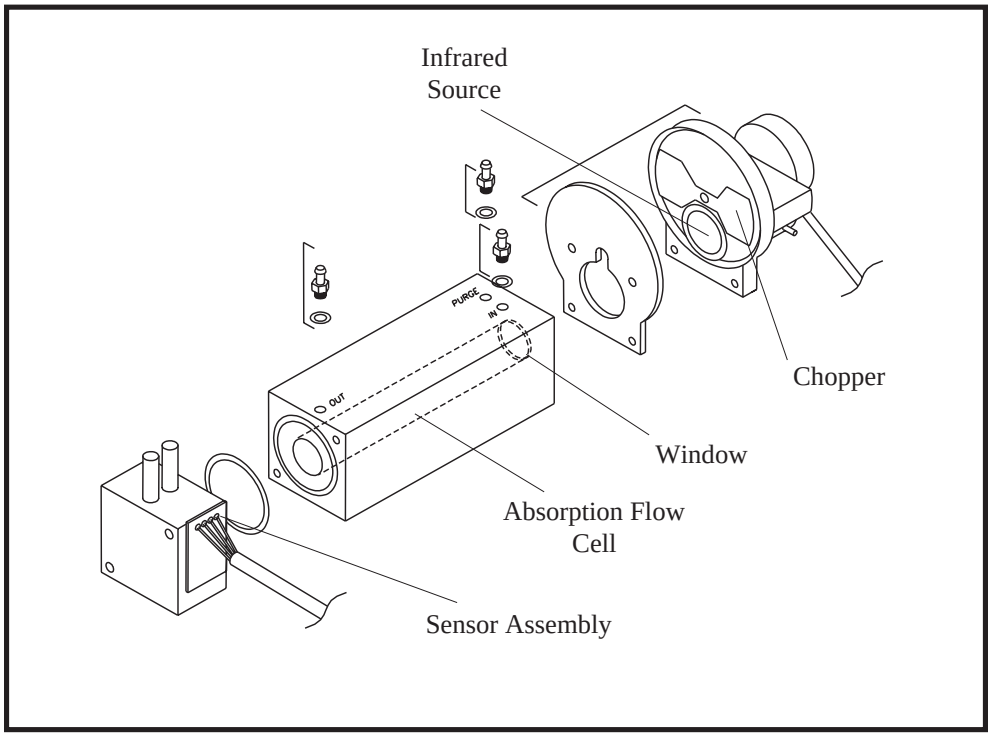


Figure 1.1. Model 1020A TOC Analyzer NDIR Detector

The source filament emits light in the infrared region of the spectrum. The infrared light passes through the measuring cell and is partially absorbed by any carbon dioxide present in the sample path. The beam then reaches the front chamber of the detector. Both the front and rear chambers of the sensor are filled with carbon dioxide. The carbon dioxide bands in the IR beam are partially absorbed in both the front and rear chambers, increasing the pressure in both chambers. The front chamber experiences a higher pressure change because of the greater amount of IR energy being absorbed. There is also a slight gas flow induced through a path connecting the two chambers.

When the absorption flow cell contains an interfering gas that possesses infrared absorption bands that overlapped with those of carbon dioxide, the interfering gas component also causes pressure pulses in the front and rear detector chambers. Selectivity is enhanced by control of the source filament temperature to ensure that overlapping interference bands (typically at 2.5 µm) have minimal effect on the detector response, which is primarily at 4.2 µm carbon dioxide band.

A chopper is placed between the source and the absorption flow cell, interrupting the source beam at 10 Hz. Pressure rises periodically in both chambers to produce a slight flow pulsation, the amplitude of which is greatest when no carbon dioxide is in the flow path. The flow pulsation is converted into an AC signal by a microflow sensor located in the path between the two chambers. AC signals are amplified and rectified to a DC voltage signal for signal processing.

Features

- Analyzes the aqueous samples for TC, TIC, TOC, and NPOC in the range of 25 ppbC to 10,000 ppmC.
- An internal, fixed volume sample loop system allows exact volumes of samples to be introduced. Sample volume is adjustable at 25 µL, 50 µL, 100 µL, 200 µL, or 400 µL.
- The combustion temperature is adjustable from 200°–950°C in one-degree increments. The optimal temperature range is 680°–900°C; higher temperatures will reduce the life of the combustion tube.
- Analyzes samples with high levels 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 400 microns in diameter) for TC, TIC, TOC, and NPOC; therefore, these samples do not need to be filtered before analysis. The combustion method allows quantitative carbon oxidation in the particulates so that an accurate value is reported.
- 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 most solvents, acids, and bases.
- The single-beam photometric system in the NDIR 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. The dual-chamber sensor effectively minimizes influence due to interfering or contaminating gas components.
- Automatic outlier recognition and reanalysis.
- All electronic and mechanical components are easily accessible for maintenance or service.
- The TOC Analyzer is controlled by a microprocessor that continually regulates temperatures, controls timing sequences, performs calculations, and monitors system diagnostics.
- Analysis results are directly printed as ppmC and µgC for TC, TIC, TOC, and NPOC for preacidified standards.

- Features singlepoint or multipoint (up to five points) calibration.
- Retains analysis conditions without external power, including calibration information and the analytical methodology. The operator can select analysis conditions or return to default conditions via files on disk.
- Conditions of analysis (times, temperatures, and volumes) can be displayed on the screen at any time.
- Automatically samples from a sample vial using the fixed volume sample loop to allow unattended replicate sampling.
- For unattended operation, an optional Model 1051 Autosampler with variable-speed stirring (88-position or 53-position) is available.
- Has several modes of analysis for a single sample, each of which can be selected by key entry. These include: TC only, TIC only, and TC/TIC/TOC. NPOC can be determined by using the Model 1051 Autosampler or externally preacidifying and prepurging the sample. POC can be determined using the POC Module option.
- A sample ID number, which increases incrementally for each successive sample, can be preset.
- Replicates and sequences of standards, samples, and check standards can be programmed and stored for unattended calibration and analysis.
- Method parameters, such as reagent volumes, TC and TIC sample inject times and detect times can be adjusted.
- WinTOC, a Windows-based software package, provides data acquisition, retrieval, postrun analysis, exporting, and storage capability from an IBM-compatible computer.
- Optional Solids TOC Analyzer can perform semiautomated analysis of soils and other solid samples.
- Optional POC Module for purgeable organic carbon (POC) analysis.

Specifications

Performance Specifications

Range*
- 25 ppb–10,000 ppmC

Precision*
- TC/TIC/TOC: 2% RSD, 100 ppb–5,000 ppm
- POC: 3% RSD

Time† of Analysis
- TC/TIC/TOC: 5–7 min total
- POC: 5–7 min total

Particulates
- <400 µm

General Specifications

Dimensions
- Model 1020A: 17" H x 13.5" W x 22" D (43 cm H x 34 cm W x 56 cm D)
- With autosampler option: 34" H x 18.75" W x 26" D (86 cm H x 48 cm W x 66 cm D)

Weight
- Model 1020A: 46 lbs (20 kg)
- With autosampler option: 68 lbs (31 kg)

Analysis Modes
- TC, TIC, TOC by difference, NPOC, and POC

Method
- TC: 680°C with platinum catalyst
- TC: 900°C with zirconium packing
- TIC: acidification (H_3PO_4) and sparging
- Furnace temperature: adjustable, 200°–950°C in 1°C increments

Sample Introduction
- Fixed volume sample loops: 25 µL, 50 µL, 100 µL, 200 µL, and 400 µL
- Sipper tube
- Autosampler (14- or 40-mL vials)

Oxygen Gas Requirements
- <1 ppm CO_2, CO, and THC at 50–60 psig (345–415 kPa)
- Recommended grade: UHP grade (99.998%) or better
- Consumption: approximately 550 mL/min

Nitrogen (for POC Option) Gas Requirements
- <1 ppm CO_2, CO, and THC at 50–60 psig (345–415 kPa)
- Recommended grade: UHP grade (99.998%) or better
- Consumption: approximately 70 mL/min

Reagent Requirements
- Phosphoric acid (H_3PO_4), 5% v/v (uses 0.2 mL/sample under normal operating conditions)

Power Requirements
- 100 (±10%) VAC, 50/60 Hz
- 110–125 (±10%) VAC, 50/60 Hz
- 210–240 (±10%) VAC, 50/60 Hz (optional)

Environmental Requirements
- Temperature: 10°–40°C
- Relative humidity: <90%, noncondensing

Detector
- Nondispersive infrared cell
- Path length: 4.3" (11 cm)

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

Options
- WinTOC (Windows-based) software
- Solids TOC Analyzer
- POC Module
- Model 1051 Vial Autosampler with variable-speed stirring (53- or 88-position)
- Printer (dot matrix)

* The range and precision of analysis is affected by sample introduction, sample size, cleanliness of sample containers, reagent purity, gas purity, and good laboratory practices.
† Time is dependent on sample properties and may vary with the matrix.

Safety Information

The OI Analytical Model 1020A TOC Analyzer meets the following International Certification when tested in typical configuration:

LVD 73/23/EEC:1974
IEC 1010-1: 1990 + A1/EN 61010-1: 1993
CSA C22.2 No. 1010.1 - 92
UL 3101, 1st Ed.

The Model 1020A TOC Analyzer also meets the following Electromagnetic Compliance Certification:

EMC. **Directive 89/336/EEC: 1989**
EN 50082-1: 1992
CISPR 11:1990/EN55011 (1991) Group 1 Class A
IEC 801-2/EN61000-4-2
IEC 801-3/EN61000-4-3
IEC 801-4/EN61000-4-4
IEC 801-4/EN61000-4-6

The Model 1020A TOC Analyzer 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 TOC Analyzer has been compromised, disconnect the instrument from all power sources and secure the instrument against unintended operation.

The exposure to personal hazards for the TOC Analyzer 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 to maintain the TOC Analyzer 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 TOC Analyzer 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 faulty or frayed power cords.
- Perform periodic leak checks on supply lines, fittings, and pneumatic plumbing.
- Arrange gas lines so that they cannot 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 to the underside of the unit.
- Wear safety glasses to prevent possible eye injury.
- Do not replace blown fuses inside the TOC Analyzer. Only trained service personnel should access the right bay of the TOC Analyzer.

- Do not perform unauthorized modifications or substitute parts that are not OI Analytical original parts. Any unauthorized modifications or substitutions may 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 associated with TOC analysis has 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 equipment used in TOC analysis.

- Hydrochloric acid and phosphoric acid have been identified as corrosive and toxic materials. Pure material 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 handling any materials containing these substances.

- 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. Avoid exposure to fumes or dust.

- Oxygen and compressed air have been classified as oxidizers. These gases and their compressed gas cylinders 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. 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:

Caution or Warning: See accompanying instruction.

Indicates a hot surface.

Indicates hazardous voltages.

Indicates earth (ground) terminal.

Definitions

Water analysis employs its own language, complete with a wide range of abbreviations and acronyms. The understanding of TOC analysis depends on a familiarity with the terms below.

TC - Total Carbon: TC is the sum of all of the carbon present in a sample, regardless of the phase or functionality. TC may also be expressed as the sum of the total inorganic carbon and the total organic carbon.

TIC - Total Inorganic Carbon: TIC is the total amount of the carbon dioxide in the solution released following sample acidification. Due to the aqueous dissociation of carbon dioxide, inorganic carbon (IC) is the total carbon that exists in solution as carbonate ion, bicarbonate ion, and carbon dioxide.

TOC - Total Organic Carbon: TOC is all of the organic carbon in a sample, not including carbon dioxide or its dissociation products.

SOC - Suspended Organic Carbon: SOC, also known as particulate organic carbon, is organic carbon that has a particle size greater that 0.45 µm.

DOC - Dissolved Organic Carbon: DOC is organic carbon content of a sample that remains following the filtration of a sample through a 0.45-µm filter.

NPOC - Nonpurgeable Organic Carbon: NPOC is organic carbon that remains in solution after a sample has been sparged (bubbled) by a gas stream.

POC - Purgeable Organic Carbon: POC is organic carbon that may be purged from solution by a gas stream.

TC Blank: This value is a measure of the overall background carbon level in the combustion furnace and can be a general indication of the low-level performance of the catalyst. The TC blank value is subtracted from the raw area counts for sample mass calculations.

Offset: This value is a measure of the overall background carbon level in the combustion furnace plus the trace carbon content in reagent water used in the preparation of standards and check standards. It is calculated as the y-intercept of the calibration curve and is subtracted from the raw area counts for check standard mass calculations. The offset value, due to how it is calculated, includes the contribution from the TC blank. For this reason, the TC blank is not used separately to calculate the mass of a check standard.

Standard: A standard is a sample with a known amount of added carbon and is used to calibrate the TOC Analyzer. The trace concentration of carbon in the reagent water, which is used to prepare standards, can be accounted for (in the offset value) by including a zero-point calibration standard (using reagent water) in the calibration curve.

Check Standard: A check standard is a standard that is analyzed to confirm the calibration. The Model 1020A TOC Analyzer will subtract the offset value from the check standard area to compensate for the contribution of reagent water to the standard value.

Method Summary

Total Carbon (TC) is determined by measuring the carbon dioxide produced by complete oxidation of all carbon present in a sample. The sample is typically combusted in an oxygen-rich atmosphere at 680°C (some applications require combustion at 900°C). The resulting carbon dioxide is detected by the nondispersive infrared (NDIR) detector.

Total Inorganic Carbon (TIC) is determined by measuring the carbon dioxide produced following sample acidification. Acidification lowers the sample pH, thereby forcing the equilibrium of the dissociation products of bicarbonate and carbonate ions to carbon dioxide. The inorganic carbon dioxide is then purged from the sample and detected by the NDIR detector.

Total Organic Carbon (TOC) is determined by taking separate TC and TIC measurements and subtracting the TIC from the TC. The result is the true TOC value, inclusive of the POC.

Purgeable Organic Carbon (POC) is determined by measuring the carbon dioxide produced following sample acidification, sparging, and combustion. Acidification of the sample releases the inorganic carbon in the form of carbon dioxide. The POC fraction of the sample and the inorganic carbon dioxide are transferred to the POC Module, where the POC fraction is collected on the trap. The carbon dioxide is not trapped and continues to the NDIR detector, where TIC is detected. The trap is then heated, releasing the POC fraction. The POC is then transferred to the furnace, where all the carbon is combusted to carbon dioxide. This carbon dioxide fraction is then measured by the NDIR detector to compute the POC concentration.

Interferences

Because carbon is ubiquitous, reagents, water, and glassware cannot be cleaned completely of it. Method interference (positive bias) may be caused by contamination of the gas, dilution water, reagents, glassware, processing hardware, or other sources. Good laboratory practices must be employed when preparing standards or glassware to avoid such interference. It is also important to use the highest purity standards, gases, and water when performing TOC analysis.

The infrared detector is employed in the TOC Analyzer because of its selectivity to carbon dioxide. Compounds other than carbon dioxide, such as O_2, HCl, SO_2, NO_2, and O_3, will not interfere with the detector, making it the ideal choice for samples that may contain chemicals that might interfere with other methods of detection.

Water may interfere with the response of the detector. It is important to maintain a water-free environment to the NDIR detector. This is accomplished automatically by passing the carbon dioxide stream through a permeation tube before it reaches the NDIR detector. For proper care of the NDIR detector, this tube should be replaced periodically, and gas flow must be maintained through the instrument.

Reagents

The Model 1020A TOC Analyzer requires acid for the analysis of TIC. Other reagents are convenient as standards for specific analyses. All standards are available from OI Analytical at (800) 653-1711 or (979) 690-1711.

Gas: The gas source must be free of carbon dioxide and carbon species for accurate reproducible analysis. High purity gas with appropriate water and carbon dioxide traps is recommended.

Reagent Water: The water used for blanks and for standard development should contain as little carbon as possible. It is recommended that reagent water contain <200 ppb total carbon. Appropriate water purification systems are required for accurate and reproducible analyses.

WARNING: *Phosphoric acid and hydrochloric acid are corrosive and harmful. Use appropriate care when handling.*

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

Hydrochloric Acid (5% vol/vol): A 5% hydrochloric acid solution is prepared by adding 13.8 mL of Acid Reagent Grade (36%) HCl to reagent water to create a total volume of 1 L.

Potassium Biphthalate (KHP) (1,000 ppmC TOC stock solution): A 1,000 ppm carbon (ppmC) as KHP standard is prepared by adding 2.125 g of KHP to reagent water to a total volume of 1 L. KHP is hygroscopic and must be dried in an oven at 110°C to a constant mass and cooled in a desiccator before weighing. The KHP stock solution has a shelf life of approximately three weeks.

Sodium Carbonate (Na_2CO_3) (1,000 ppmC TIC stock solution): A 1,000 ppmC as Na_2CO_3 standard is prepared by adding 8.824 g of Na_2CO_3 to reagent water to create a total volume of 1 L. The Na_2CO_3 stock solution has a shelf life of approximately three weeks.

Chapter 2
Description of Components

Model 1020A - Front View

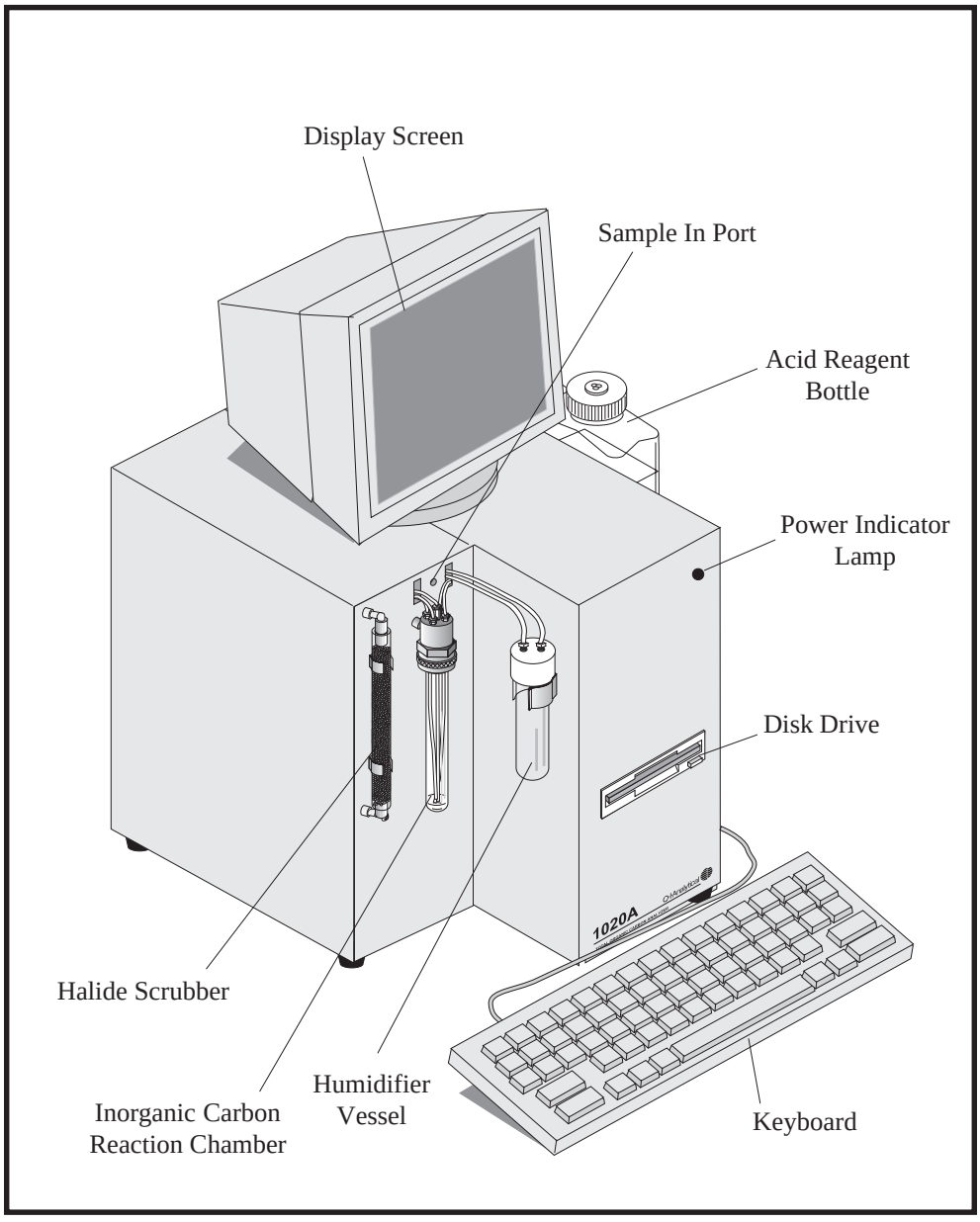


Figure 2.1. Model 1020A TOC Analyzer - Front View

Acid Reagent Bottle is a reservoir for the reagent, which should be monitored according to the number of analyses and volume of reagent used per sample. See Chapter 1, “Introduction,” for preparing the reagent to add to this bottle.

Disk Drive is a standard 3.5-inch computer disk drive. The operating system for the TOC Analyzer is loaded on to the internal “chip disc” from this disk drive. The program disk must be removed from the disk drive after installing the operating system.

Display Screen is a 9-inch monochrome monitor used to view selected settings and parameters (not included with the WinTOC option).

Halide Scrubber is used to remove excess halide content, which may be corrosive to the infrared detector.

Humidifier Vessel is a vessel for holding reagent water to humidify the gas going to the combustion chamber.

Inorganic Carbon Reaction Chamber (IC Chamber) is the chamber where the sample is acidified and purged to determine the TIC content.

Keyboard is used to enter settings and select parameters displayed on the display screen (not included with the WinTOC option).

Power Indicator Lamp is illuminated when the TOC Analyzer’s power is turned on.

Sample In Port 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.

Model 1020A - Back View

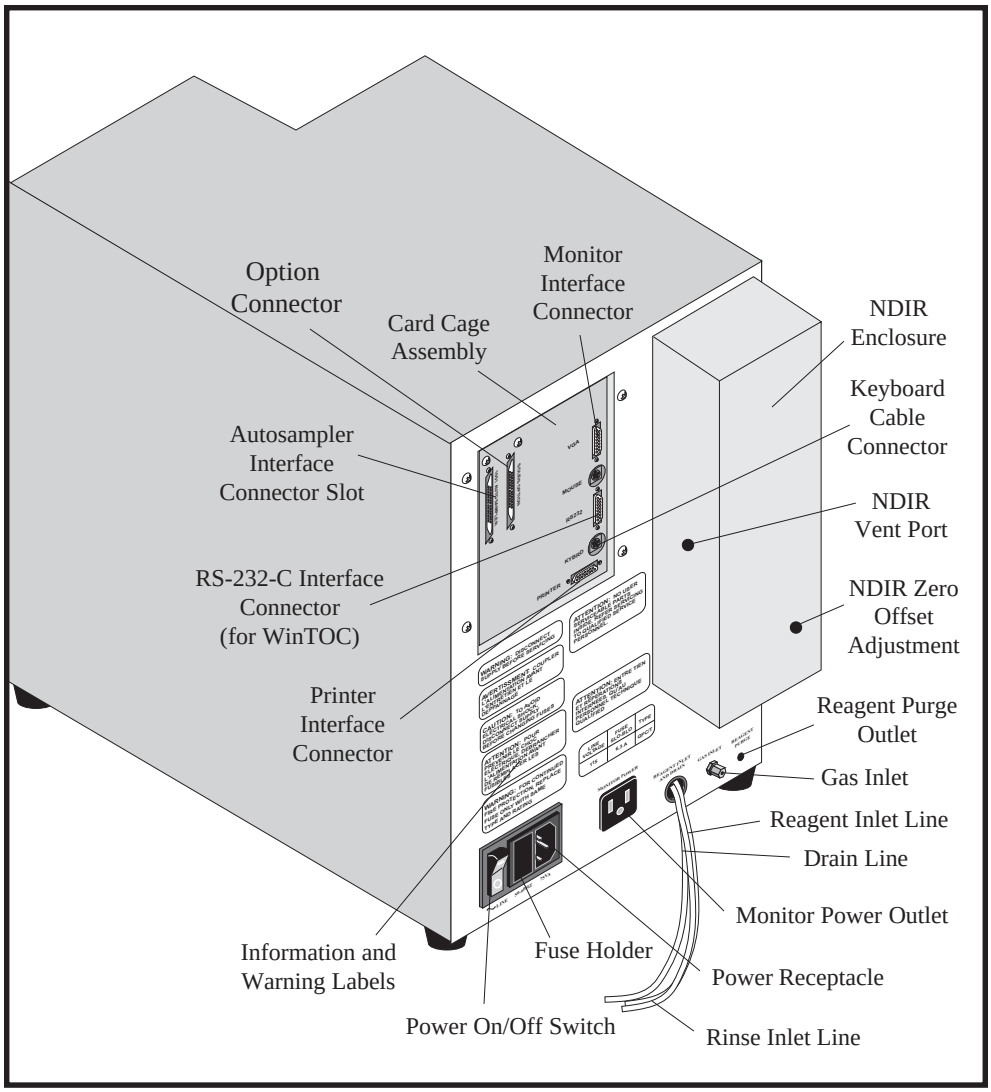


Figure 2.2. Model 1020A TOC Analyzer - Back View

Autosampler Interface Connector Slot allows the Model 1051 Autosampler to interface to the Model 1020A via an interface connector and cable. If the TOC Analyzer does not have an autosampler, this slot may be empty.

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

Drain Line provides an exit for sample that has overfilled the sample loop and allows waste from the analysis to exit the TOC Analyzer. Route this line to an appropriate waste receptacle.

Fuse Holder houses the main fuse, which protects the TOC Analyzer from over-current operation.

Gas Inlet provides an inlet for compressed 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 TOC Analyzer and inform the operator of voltage requirements.

Keyboard Cable Connector connects the TOC Analyzer to the keyboard.

Monitor Interface Connector connects the monitor to the TOC Analyzer.

Monitor Power Outlet allows output power for the TOC Analyzer's monitor.

NDIR Enclosure houses the NDIR detector.

NDIR Vent Port is the exhaust outlet from the NDIR detector.

NDIR Zero Offset Adjustment is used to adjust the signal level or baseline of the NDIR detector.

Option Connector connects the TOC Analyzer to either the Solids TOC Analyzer or POC Module Option.

Power On/Off Switch is the power control switch. A power-up self test accompanies turning on the TOC Analyzer.

Power Receptacle connects the TOC Analyzer 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.

Reagent Inlet Line is the inlet line for acid reagent from the acid reagent bottle to the TOC Analyzer.

Reagent Purge Outlet provides gas to purge the acid reagent in the reagent bottle.

Reagent Rinse Line is the inlet line from the rinse bottle to the TOC Analyzer.

RS-232-C Interface Connector allows interface to an IBM-compatible personal computer running WinTOC (not used with Firmware operation). This connector interfaces with a standard 9-pin male connector.

POC Module

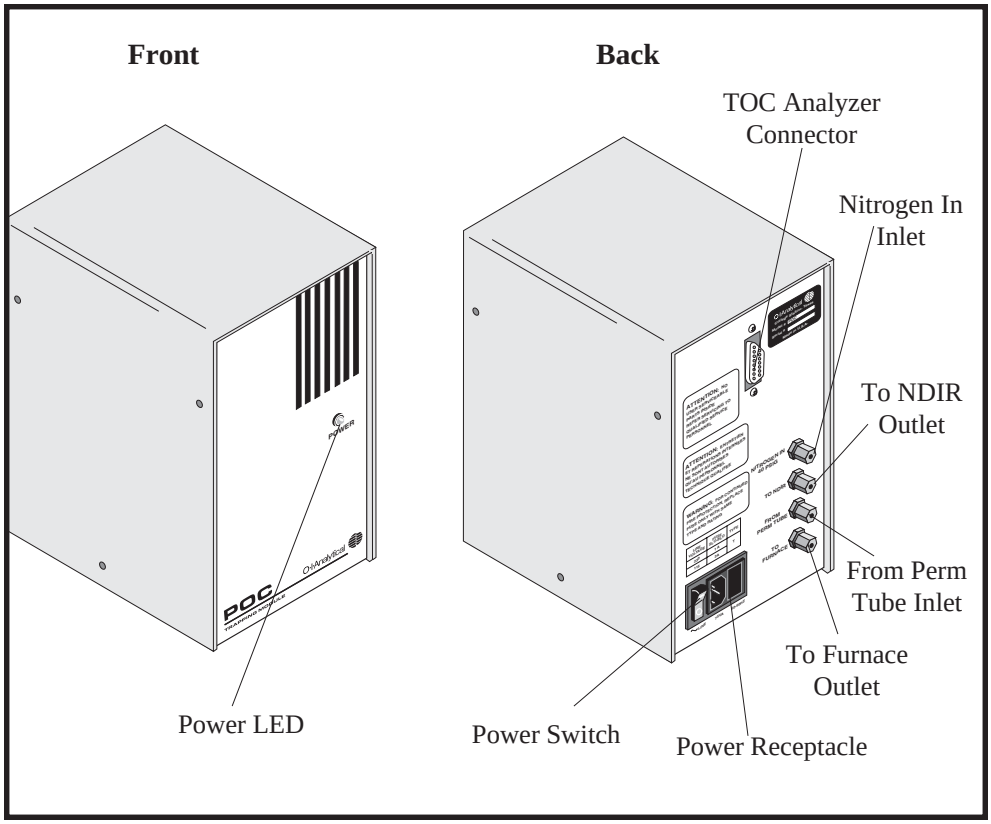


Figure 2.3. POC Module - Front and Back Views

From Perm Tube Inlet connects the gas tubing (clear) from the Model 1020A permeation tube to the POC Module.

Nitrogen In Inlet connects the nitrogen supply (carrier gas) (approx 40 psi) to the POC Module.

Power LED indicates the power status.

Power Receptacle is an IEC (International Electrotechnics Convention)-type power inlet receptacle. It connects the POC Module to an appropriate power source via a power cable.

Power Switch (rocker switch) turns the power to the POC Module on/off. Power status is indicated by the Power LED on the front of the POC Module.

To Furnace Outlet connects CO_2 tubing (yellow) from the POC Module to the Model 1020A furnace.

To NDIR Outlet connects CO_2 tubing (yellow) from the POC Module to the NDIR detector.

TOC Analyzer Connector (15-pin connector) connects the POC Module to the TOC Analyzer.

Notes

Chapter 3
Installation

In Chapter 2, “Description of Components,” the names and functions of the various Model 1020A TOC Analyzer components were outlined. These names refer to components involved in the installation of the Model 1020A.

The OI Analytical Model 1020A TOC Analyzer is supplied as a stand-alone system. The installation and operation of the TOC Analyzer is described in this manual for autonomous operation. Accessories, including autosampler, Solids TOC Analyzer, POC Module, and WinTOC software, are sold separately. Installation of accessories, except the POC Module, is described in their respective operating manuals.

Note: To install and configure WinTOC (Windows-based) software, refer to the *WinTOC 1020A Operator’s Manual.*

Site Preparation

Environmental Considerations

The Model 1020A TOC Analyzer requires gas and electrical connections within close proximity. This instrument is designed to operate under standard laboratory conditions with temperatures ranging from 10°– 40°C (50°–104°F) and a relative humidity of <90%, noncondensing. The TOC Analyzer has a footprint of 13.5" x 22" (34 cm x 56 cm) and a weight of 46 lbs (20 kg). The TOC Analyzer and autosampler unit have a combined footprint of 18.75" x 26" (48 cm x 66 cm) and a weight of 68 lbs (31 kg).

External Requirements

The Model 1020A TOC Analyzer has certain requirements that should be taken into consideration before beginning the installation procedure.

- Fittings and supply lines enter through the back of the instrument. Place the TOC Analyzer so that the back panel can be accessed.
- The supplied power cord is 72" (180 cm) long. Place the TOC Analyzer in close proximity to an appropriate power supply.
- The TOC Analyzer contains a microprocessor that requires surge protection (not included).

- The Model 1020A TOC Analyzer requires high purity oxygen gas for proper operation. To easily regulate the gas to a constant flow, place an on/off valve (not included) before the inlet to the TOC Analyzer.

- The TOC Analyzer requires a drain receptacle at a lower level than the instrument. A permanent drain is preferred; however, a waste bottle can be used and emptied periodically.

- Gas flow rates must be periodically checked or adjusted during installation and maintenance. An accurate gas flow meter is necessary; a digital flowmeter is recommended.

Initial Setup

Follow the steps below to unpack and prepare the TOC Analyzer for installation.

1. Remove the instrument from the shipping boxes and check the items against the component list. If any item is damaged, notify the carrier immediately. Save all packing materials until proper operation of all components is 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 and with the original packing material. If instruments are damaged due to improper shipping to OI Analytical, OI Analytical will not be responsible for cost of repairs. If proper shipping materials are not available, contact the Order Entry Department at (800) 653-1711 or (979) 690-1711.

2. Place the TOC Analyzer on a bench near the gas and electrical sources. The power cord is 72" (180 cm).

3. Connect one end of the power cord to the power receptacle on the back of the TOC Analyzer. Connect the other end of the power cord to an appropriate power supply.

Required Materials

The following materials will be required during the installation process.

- ¼" open-end wrench
- 7⁄16" open-end wrench
- ⅛" tubing cutter
- High purity oxygen source
- Oxygen regulator
- ⅛" O.D. x 0.0063" I.D. Teflon® gas tubing
- 5% phosphoric acid reagent (See "Reagents" in Chapter 1.)
- Reagent water (<200 ppb TOC)

Interior Connections

Follow the steps below to remove the covers of the instrument before making interior connections.

WARNING: *Hazardous voltages can exist inside the TOC Analyzer. Use extreme caution when accessing the inside of the unit.*

1. Remove the left panel of the instrument by removing the Phillips-head screws on the top and left side of the instrument.

2. Lift the cover straight up and off. Refer to Figure 3.1 for the major flow components of the Model 1020A TOC Analyzer.

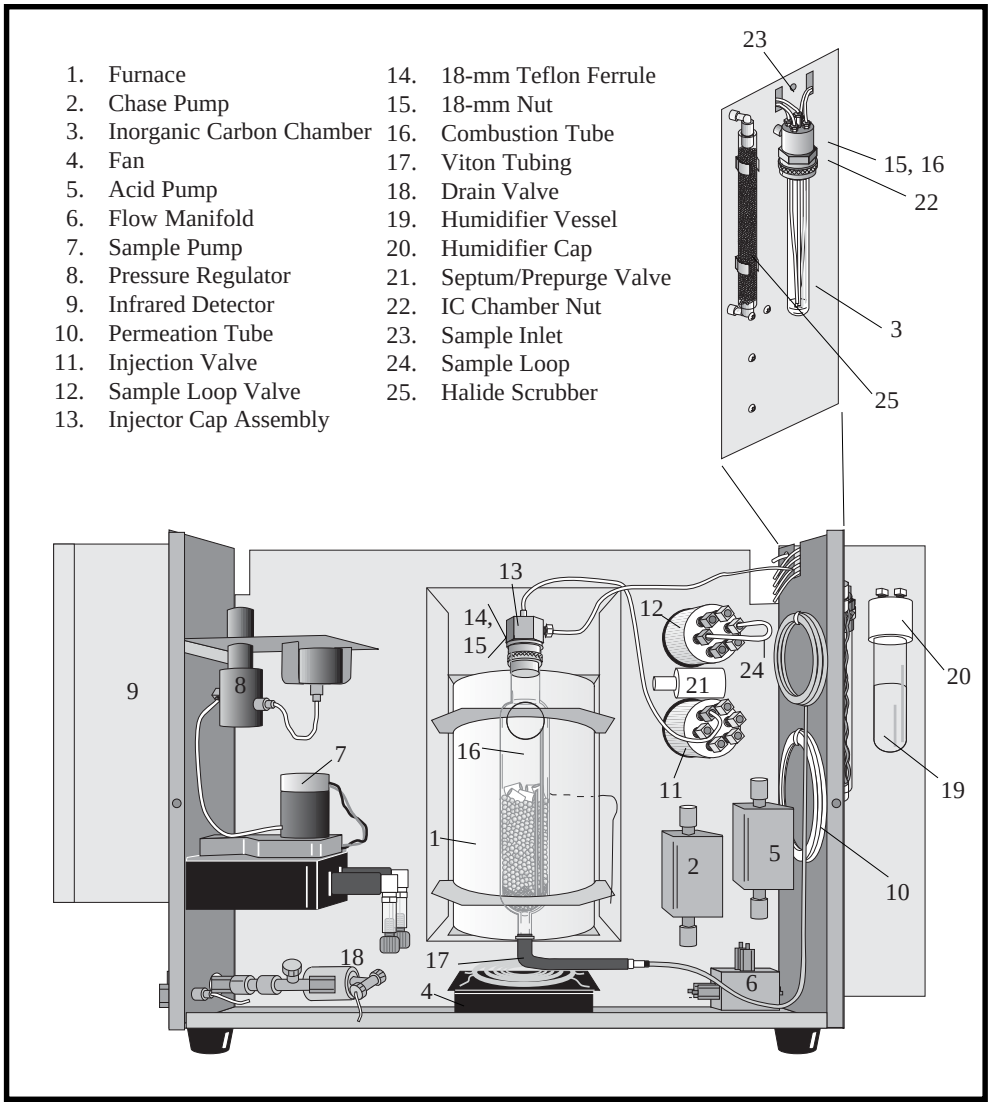


Figure 3.1. Left Interior of the Model 1020A TOC Analyzer

Sample Loop Installation

Follow the steps below to install a sample loop.

1. Locate the sample valve on the inside of the instrument (Figure 3.1, #12).

2. Verify the volume of the sample loop by reading the value on the tag.

3. Record the value written on the 25-, 50-, 100-, 200-, and 400-µL sample loops.

   _____ µL
   _____ µL
   _____ µL
   _____ µL
   _____ µL

4. To install a different sample loop than the one in the unit, unscrew the loop from positions 3 and 6 on the sample valve and remove the loop. See Appendix A, "Selecting a Sample Loop," to choose the correct sample loop.

CAUTION:
Overtightening the loop ends can cause the sample loop to collapse.

5. Place the screw connectors and front and back ferrules on the sample loop, ensuring that the Teflon loop does not extend beyond the flat part of the ferrule.

6. Place the loop ends into positions 3 and 6 and secure to *finger-tight*.

Combustion Tube Installation

900°C Operation

1. Locate the combustion tube (Part #279067), platinum gauze (Part #281121), fill tube (Part #303453), funnel, zirconia (Part #319433), and quartz (Part #303024) from the start-up kit.

2. Loosen the four screws that hold the furnace heat shield in place, and remove the shield by lifting up and sliding down and out.

3. Using the fill tube, fit a piece of platinum gauze into the bottom of the combustion tube.

4. Insert the fill tube into the combustion tube. Using the funnel, pour approximately 2 cm of zirconia into the fill tube (Figure 3.2A).

5. Lift the fill tube slowly and allow the zirconia to fill the combustion tube (Figure 3.2B). Continue to add zirconia into the combustion tube until the zirconia reaches a level of 10.25 cm (4") from the top of the combustion tube (Figure 3.2C).

6. Place another piece of platinum gauze on top of the zirconia.

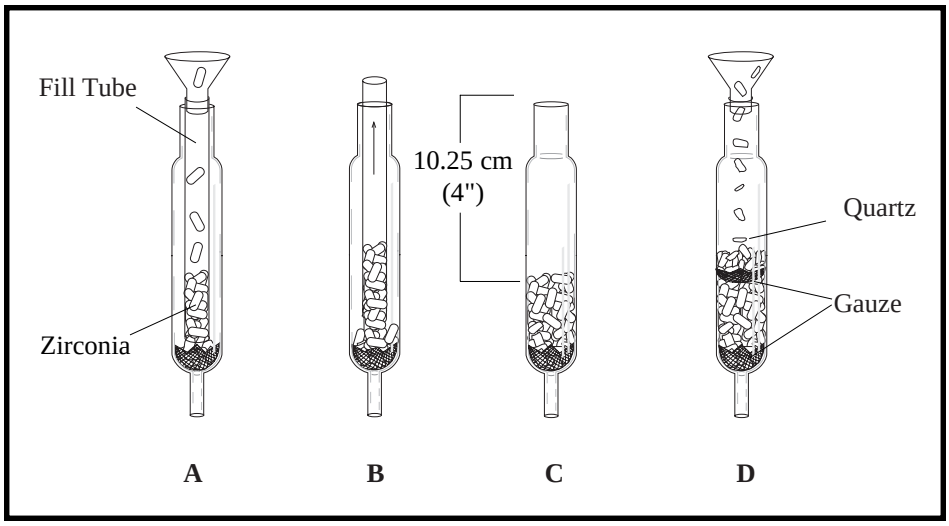


Figure 3.2. Combustion Tube Filling Procedure for 900°C Operation

7. Using the funnel, pour 1.0 cm (0.4") of quartz on top of the platinum gauze.

8. Verify that both platinum gauze are in place on the finished combustion tube (Figure 3.2D).

9. Gently insert the filled combustion tube into the top of the furnace. Place a few drops of reagent water in the end of the Viton® tubing (Part #313908). Fit the Viton tubing onto the bottom of the combustion tube.

10. Using a pair of pliers, slide the spring clamp (Part #313890) over the Viton tubing. Place the clamp on the end of the Viton tubing, allowing the spring clamp to hold the Viton tubing onto the bottom of the combustion tube.

11. Place a few drops of reagent water on the end of the adapter fitting (Part #313916) and slide it into the Viton tubing. See Figure 3.3.

12. Attach the coupling fitting (Part #166274) to the adapter fitting. Place the ferrule (Part #317545) on the end of the yellow fitting (Part #319346) on the yellow combustion tubing. Screw the yellow fitting and ferrule into the coupling fitting. (See Figure 3.3)

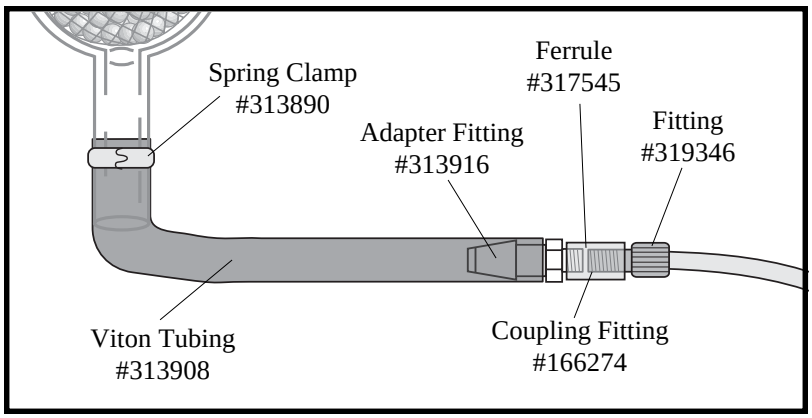


Figure 3.3. Combustion Tube Outlet Assembly

13. Attach the 18-mm nut and the 18-mm Teflon ferrule to the top of the combustion furnace, facing up. Attach the injector cap to the nut and ferrule.

14. Replace the combustion furnace heat shield.

15. Leave the cover off of the TOC Analyzer until gas connections are completed and flow is established.

680°C Operation

1. Locate the combustion tube (Part #279067), platinum gauze (Part #281121), fill tube (Part #303453), funnel, catalyst (Part #303032), and quartz (Part #303024) from the start-up kit.

2. Loosen the four screws holding the furnace heat shield in place. Remove the shield by lifting up and sliding down and out.

3. Using the fill tube, fit the gauze mesh into the bottom of the combustion tube.

4. Insert 0.5" (1.27 cm) of quartz wool (Part #144501) on top of the mesh.

5. Insert the fill tube into the combustion tube. Using the funnel, pour approximately 2 cm of catalyst into the fill tube (Figure 3.4A).

6. Lift the fill tube slowly, allowing the catalyst to fill the combustion tube (Figure 3.4B). Continue to pour catalyst into the combustion tube until the catalyst reaches a level of 10 cm from the top of the combustion tube (Figure 3.4C).

7. Using the funnel, pour approximately 1 cm of quartz on top of the catalyst bed (Figure 3.4D).

8. Verify that the gauze and wool are in place on the finished combustion tube.

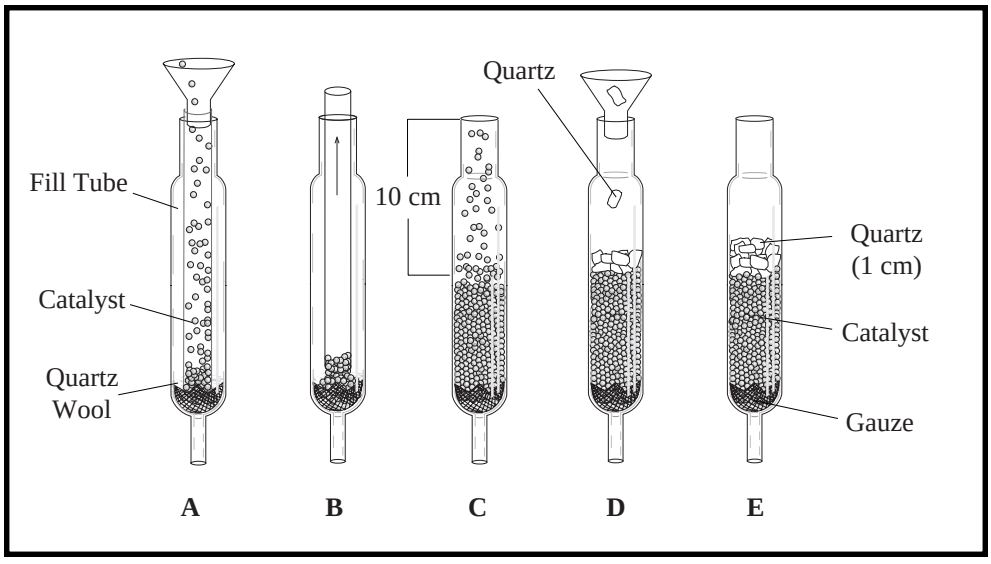


Figure 3.4. Combustion Tube Filling Procedure for 680°C Operation

9. Gently insert the filled combustion tube into the top of the furnace. Place a few drops of reagent water in the end of the Viton tubing (Part #313908). Fit the Viton tubing onto the bottom of the combustion tube.

10. Using a pair of pliers, insert the spring clamp (Part #313890) over the Viton tubing. Place the clamp on the end of the Viton tubing, allowing the spring clamp to hold the Viton tubing onto the bottom of the combustion tube.

11. Place a few drops of D.I. water on the end of the adapter fitting (Part #313916) and slide it into the Viton tubing. See Figure 3.3.

12. Attach the coupling fitting (Part #166274) to the adapter fitting. Place the ferrule (Part #317545) on the end of the yellow fitting (Part #319346) on the yellow combustion tubing. Screw the yellow fitting and ferrule into the coupling fitting. See Figure 3.3.

13. Attach the 18-mm nut and the 18-mm Teflon ferrule to the top of the combustion furnace, facing up. Attach the injector cap to the nut and ferrule.

14. Replace the combustion furnace heat shield.

15. Leave the cover off of the TOC Analyzer until gas connections are completed and flow is established.

Back Panel Connections

1. Attach the line from the regulator to the Gas Inlet fitting on the back of the TOC Analyzer. It is recommended for convenience to install a shutoff valve and an appropriate carbon dioxide trap between the tank and the instrument inlet.

2. Attach the keyboard cable into the connector labeled “Keyboard” on the TOC Analyzer.

3. Attach the monitor cable into the connector labeled “Video” on the TOC Analyzer.

4. Attach the printer cable into the connector labeled “Printer” on the TOC Analyzer.

5. Remove the black drain line from the back of the TOC Analyzer and route it to either a drain bottle or a waste bottle.

Note: Although the Model 1020A uses a peristaltic pump to drain, the drain/waste bottle must be lower in elevation than the TOC Analyzer to prevent the possibility of waste siphoning back into the TOC Analyzer.

6. Locate the red ⅛" reagent inlet line from the back of the instrument and route it through the appropriate hole in the supplied acid bottle cap.

7. Locate the clear ⅛" reagent rinse line from the back of the instrument and route it through the appropriate hole in the rinse bottle cap.

8. Attach the clear 1⁄16" reagent purge line to the Reagent Purge outlet on the back of the TOC Analyzer. Route one end to the appropriate inlet on the acid bottle and the other end to the appropriate hole in the rinse bottle cap.

9. Fill the acid bottle with the 5% phosphoric acid reagent (see “Reagents” in Chapter 1).

10. Attach the cap and place the acid bottle in the reagent tray on the right side of the TOC Analyzer.

11. Fill the rinse bottle with reagent water and attach the cap.

Front Panel Connections

1. Fill the humidifier vessel to ¾-full with reagent water, add one pellet of sodium hydroxide, and snap the vessel into position (see Figure 3.1).

2. Remove the plug from the “Sample In” port and attach the sipper tube.

Installing the Inorganic Carbon (IC) Chamber

1. Locate the IC chamber (Part #199521) in the start-up kit.

2. Remove the packing from the four Teflon tubes that are suspended from the 18-mm fitting on the TOC Analyzer front panel.

3. Insert the Teflon tubes into the IC chamber.

4. Insert the open end of the chamber into the 18-mm fitting on the front panel of the TOC Analyzer.

5. Hand-tighten the 18-mm fitting.

6. Verify that the ends of the red tubing and the black tubing are at the corresponding colored lines on the IC chamber. The tubing should not go below these lines.

Flow Settings

1. Turn on the oxygen at the regulator and set it to 50–60 psig.

2. Ensure that the pressure regulator in the TOC Analyzer is set to 18 psi.

3. Check for leaks on the external gas connections using a leak detector (e.g., Snoop®).

4. Reattach the left side of the TOC Analyzer and secure the panel with the previously removed screws.

5. Place the monitor on top of the instrument, and place the keyboard in front of the unit as shown in Figure 2.1.

Installing the POC Module Option

1. Verify that the Model 1020A is turned off.

2. Remove the left bay cover of the Model 1020A.

3. Place the POC Module on the left side of the TOC Analyzer.

4. Connect one end of the Options cable to the 15-pin connector on the back of the POC Module (see Figure 2.3). Connect the other end of the cable to the Option connector on the back of the TOC Analyzer.

5. Attach the nitrogen line to the Nitrogen In inlet fitting on the back of the POC Module.

Note: Refer to Figure 3.5 for steps 6–10.

6. Locate the yellow tubing from the halide scrubber to the permeation tube. Cut the tubing. Attach the 1/16" female Luer adapter (Part #196386) to the section attached to the halide scrubber. Attach the 1/16" male Luer adapter (Part #194415) to the section attached to the permeation tube.

7. Two pieces of yellow tubing (Part #319329) are provided in the POC start-up kit (Part #317842). Attach one end of one of the yellow tubing to the To NDIR outlet fitting on the back of the POC Module. Route the tubing through the back of the TOC Analyzer, and attach the tubing to the adapter (Part #196386) leading to the halide scrubber.

8. Attach one end of the remaining yellow tubing to the From Perm Tube inlet fitting on the back of the POC Module. Route the tubing through the back of the TOC Analyzer, and attach the tubing to the adapter (Part #194415) leading to the permeation tube.

9. Install the tee fitting provided (Part #279125) on the clear tubing connecting the furnace to the humidifier vial.

10. Attach one end of the 42" clear tubing (Part #147901) provided to the To Furnace outlet fitting on the back of the POC Module. Route the tubing through the back of the TOC Analyzer, and attach the tubing to the tee fitting installed in step 9.

11. Replace the left bay cover on the TOC Analyzer.

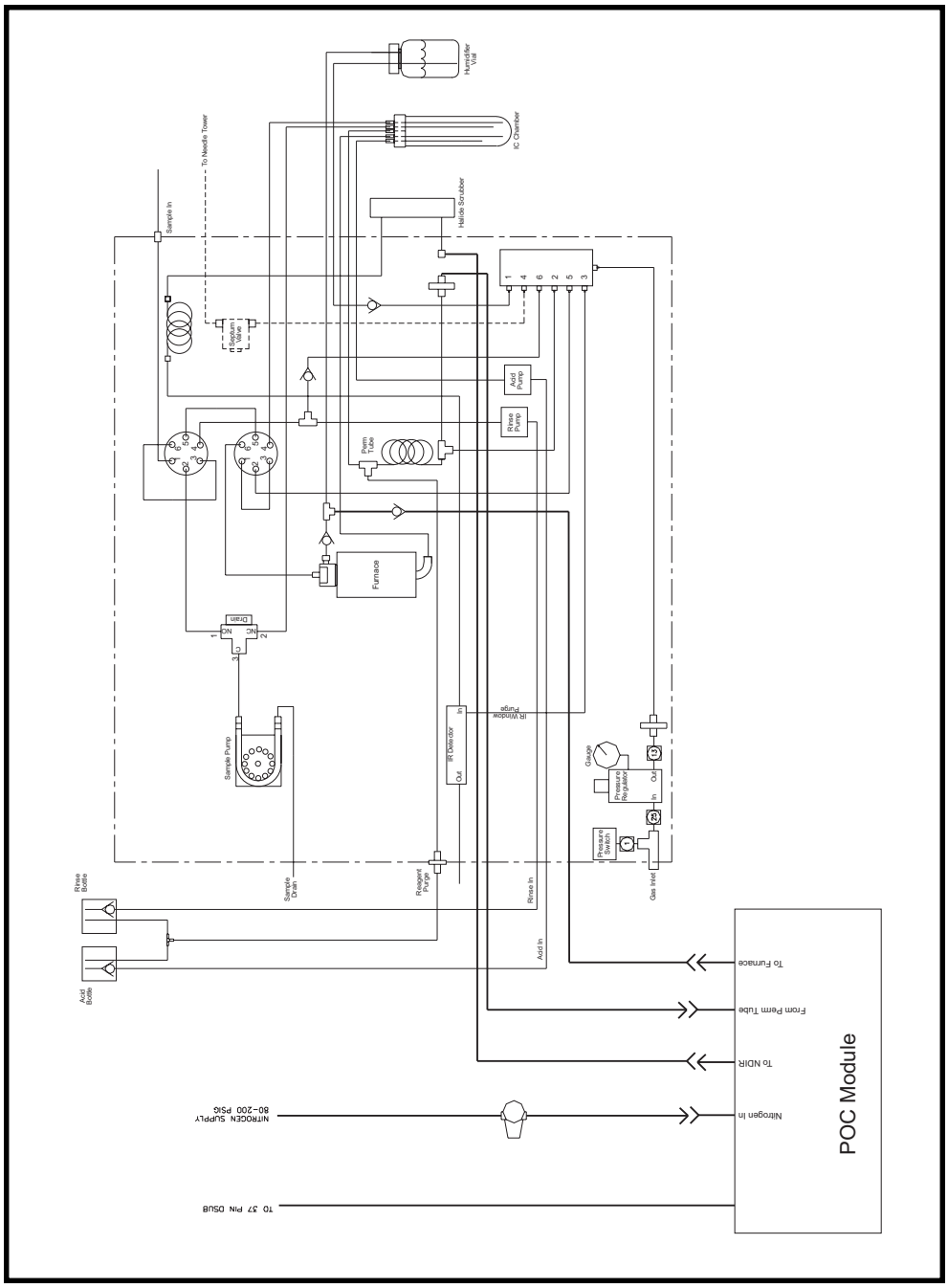


Figure 3.5. Model 1020A TOC Analyzer with POC Module Flow Diagram

Adjusting the POC Module Flow

1. Start up the Model 1020A. See “Starting Up the Unit” in Chapter 5.
2. Set the nitrogen pressure to the POC Module to approximately 40 psi.
3. Using the keyboard, press [F7] to access the DIAGNOSTICS SCREEN.
4. Use the arrow keys to select **POC Valves**. Use the [Page Up] and [Page Down] keys to select **Desorb**.
5. Remove the clear tubing from the To Furnace outlet on the back of the POC Module.
6. Connect a flowmeter to the To Furnace outlet and verify that the flow is 65–75 mL/min. If the flow is outside this limit, use the pressure regulator on the Nitrogen In tubing to adjust the flow rate.
7. Once the flow has been set, reconnect the clear tubing to the To Furnace outlet.

Notes

Chapter 4
Introduction to Firmware

This chapter provides descriptions of the menus, screens, and commands used to control the Model 1020A TOC Analyzer by keyboard and monitor using the MS-DOS®-based control program provided on the Program Disk ("Firmware"). To operate the Model 1020A TOC Analyzer, refer to Chapter 5, "Operation."

Firmware Screens

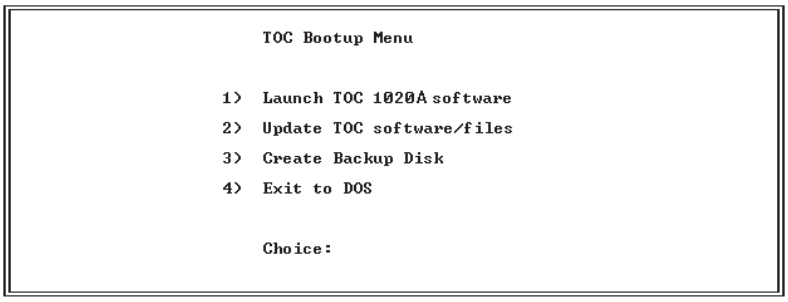
The control program consists of nine menus that are accessed by using the function keys on the keyboard. These menus can be divided into three categories: operation, configuration, and diagnostics (see Table 4.1). Each of the five screens are presented in detail in this chapter.

Table 4.1. Menus and Keystrokes for Firmware

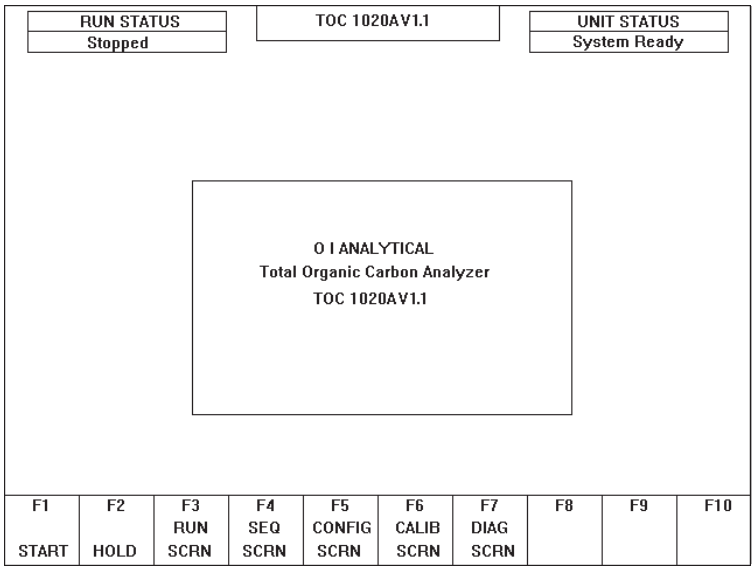
Category	Key	Function/Screen
Operation	[F1]	Start/resume the run
	[F2] (pressed once)	Pause the run or sequence
	[F2] (pressed twice)	Stop the run or sequence
	[F3]	RUN SCREEN
	[F10]	Spiral the autosampler tray
Configuration	[F4]	SEQUENCE SCREEN
	[F5]	CONFIGURATION SCREEN
	[F6]	CALIBRATION SCREEN
Diagnostics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

Bootup Screen

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



The default selection is **1) Launch TOC 1020A software**, which will execute automatically in 10 seconds if no option is selected. To select an option, press the number of the option desired on the keyboard. If no option is selected or if **1)** is selected, the **Startup Screen** appears.



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**, and **Waiting For Start**.

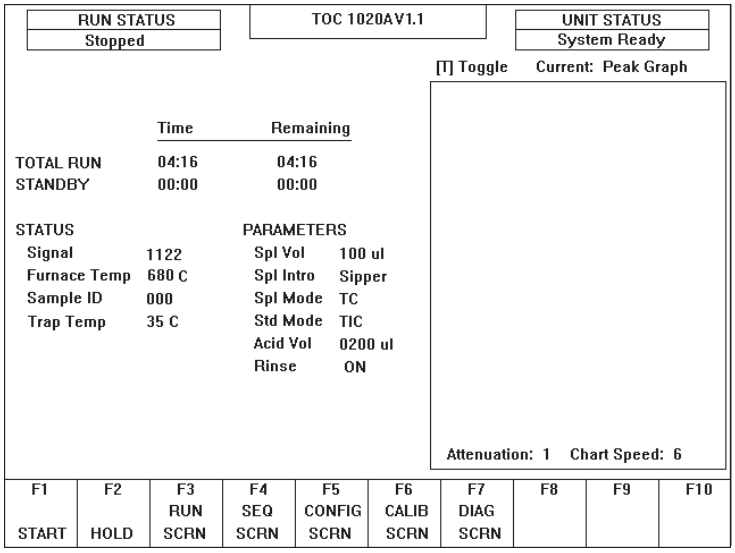
UNIT STATUS — Informs the operator if the TOC Analyzer is ready and may include errors or warnings if a problem exists with the instrument. The possibilities are **System Ready**, **Printer Error**, **Low Gas Pressure**, and others, which are discussed in Chapter 9, "Trouble-shooting."

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 SCRN) appears.



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 parameters that can be changed on this screen are **Attenuation** and **Chart Speed**. 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.

Table 4.2. Model 1020A States

State	Description
STANDBY	Idle state of the TOC Analyzer.
SAMPLE INTRO	Instrument is sampling.
TC SELECT	Valve pathway selection to the furnace.
TC INJECT	Sample is injected in the furnace.
TC DETECT	TC is converted to carbon dioxide by combustion, and the carbon dioxide from TC is detected.
TIC SELECT	Valve pathway selection to the IC chamber.
TIC INJECT	Sample is injected into the IC chamber.
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.
RINSING	Instrument is rinsing.
DRAIN	Sample and reagents are drained from the IC chamber.
CONFIG	Computing times and sequences of steps for this replicate.
RESULTS READY	Completing statistical calculations for this replicate.
UPDATING FILES	Updating the internal files.
POC Operation Only	
POC WAIT	Waiting for POC Module to signal ready for sample introduction.
POC DESORB	Desorbing compounds of interest from the POC trap.
POC TRAP COOL	Waiting for the POC trap to cool.
COLD PURGE	Nitrogen purge gas is flowing onto the POC trap.
Waiting States	
WAIT SPL	Waiting for the operator to change the sample vials with the sipper.
WAIT A/S	Waiting for the autosampler to change position.
PAUSE	TOC Analyzer is paused.

Chart Speed — The Left [←] arrow key slows down the chart speed and the Right [→] arrow key speeds up the chart speed. Only new data will be displayed at the selected chart speed; existing data will not be redrawn. The remaining parameters can be changed on other screens.

Note: The baseline of this display is automatically shifted so that the display is optimized. This shift occurs during the Standby State and appears as a vertical shift in the baseline.

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.

Furnace Temp — Displays the actual temperature of the sample furnace.

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

Trap Temp — Displays the temperature of the trap inside the POC Module.

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.

Spl Vol — Displays the volume of sample being used.

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

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

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

Acid Vol Displays the volume of acid reagent used for TIC analysis.

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

Chase/Rep Informs the operator if chase per rep is ON or OFF.

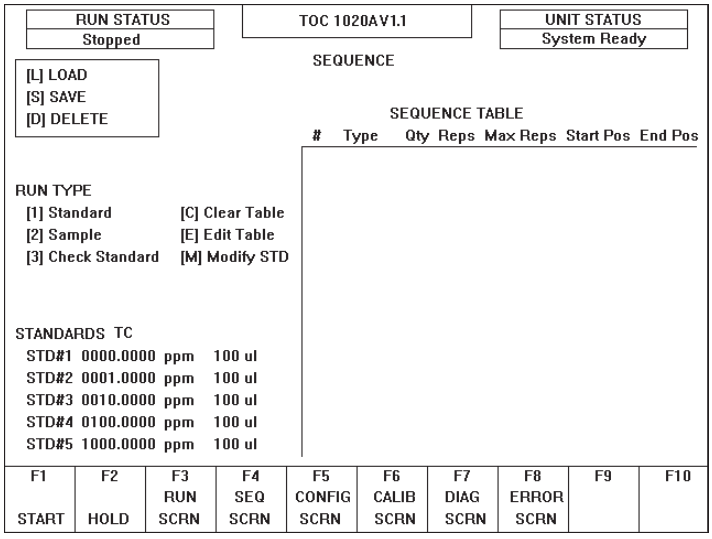
Toggle Allows the operator to change the display from graphical representation of peaks to numerical representation of results.

PAM (Preacidification module) Informs the operator if preacidification is ON or OFF (with the Model 1051 Autosampler only).

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 SCRN) appears.



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 (**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 POC) and the list of standards 1–5. The **Sample Vol** on the CONFIGURATION SCREEN is the volume of standard to be analyzed. This is the volume of the currently installed loop. These can be programmed into memory to be recalled when standards are analyzed. The TOC Analyzer can store one standard curve for each type of standard. To use a specific calibration type (i.e., TIC instead of TC), change to that standard type. Changing the standard does not affect the mode of analysis.

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.

Qty = End Pos – (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.

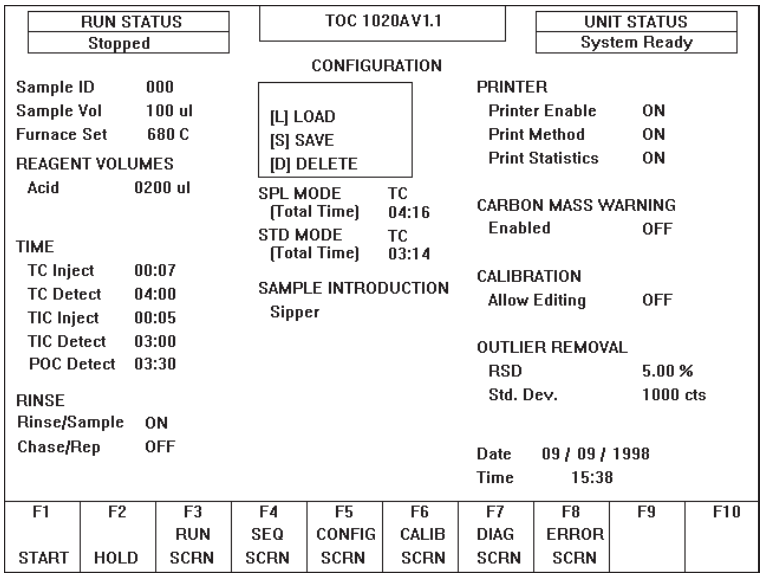
Max Reps Allows the operator to set the number of retries (0–5) the TOC Analyzer will attempt for samples that are outside of the specified reproducibility limits. The limits are defined from the CONFIGURATION SCREEN; however, the maximum number of reps for a sample is 15 and for a standard is 10. All samples, standards, and check standards in a sequence will share the same RSD and Std. Dev. criteria.

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 SCRN) appears.



The CONFIGURATION SCREEN provides access to the instrument parameters, which can then be saved as methods. Operational parameters, listed in Table 5.1, can be optimized for specific analyses.

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

Sample Vol	The volume of the sample being analyzed. The default is 100 µL. The sample volume can be changed by scrolling with the [Page Up] and [Page Down] keys. The choice of sample size is based on sample loop volumes that are entered from the DIAGNOSTICS SCREEN.
Furnace Set	Allows the operator to set the temperature of the furnace from 200°C to 950°C. The default is 680°C for operation with the platinum catalyst. For platinum catalytic operation, the temperature should not exceed 720°C.
REAGENT VOLUMES **Acid**	Allows the volume to be set between 0 µL and 2,000 µL in 100-µL increments. Default is 200 µL.
TIME	Allows the operator to set the times used for analysis.
TC Inject	Allows the operator to set the elapsed time over which the sample is injected into the combustion tube. Range of time is 00:00–1:00 min. Default is 00:10.
TC Detect	Allows the operator to set the time that the carbon dioxide from total carbon is combusted and detected. Range of time is 00:45–05:00 min. Default is 03:30.
TIC Inject	Allows the operator to set the time that the sample is injected into the IC chamber and phosphoric acid reagent is added. Range of time is 00:00–2:00 min. Default is 00:10.
TIC Detect	Allows the operator to set the time that the carbon dioxide from TIC is purged from the sample and detected. Range of time is 00:45–05:00 min. Default is 03:00.
POC Detect	Allows the operator to set the time that the POC fraction is combusted and the carbon dioxide is detected. Range of time is 00:45–5:00 min. Default is 02:00.
RINSE	Allows the rinse and chase to be set. Rinse can be programmed to occur between samples (per sample). If the **Rinse** is **OFF**, no rinses will occur. Default rinses per sample is OFF. Chases can be programmed to ocur between reps (per rep). If the **Chase** is **OFF**, no chases will occur. Default chases per rep is OFF.

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 of 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**, **TIC/POC**, and **TC/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 **POC**.

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 **Sipper** or **Autosampler**.

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.

Note: Select the sample loop volume so that the sample range falls in the middle of the concentration range of the sample loop.

Autosampler

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

Table 4.3. Approximate Detectable Range for Sample Volumes

Carbon Concentration Range	Nominal Sample Volume
5,000 –10,000 ppmC	25 µL
100–5,000 ppmC	50 µL
5–2,500 ppmC	100 µL
0.5–1,250 ppmC	200 µL
0.025–625 ppmC	400 µL

PRINTER Allows control of the printer that is connected to the TOC Analyzer.

Printer Enable Turns on printer output so information can be printed.

Note: Data is not saved on the TOC diskette and must be printed to retain a record.

Print Method Enables printing of the method as it currently exists in the TOC Analyzer.

Print Statistics Enables printing of the replicate average and standard deviation of samples and standards.

CARBON MASS WARNING Allows the enabling/disabling of the over mass warning. If **Enabled** is on and the carbon mass of the sample exceeds 275 µgC, a warning will appear on the screen. If **Enabled** is off, no warning will appear.

CALIBRATION Allows editing of standard calibration information. If **Allow Editing** is off, standards may not be deleted and vice versa.

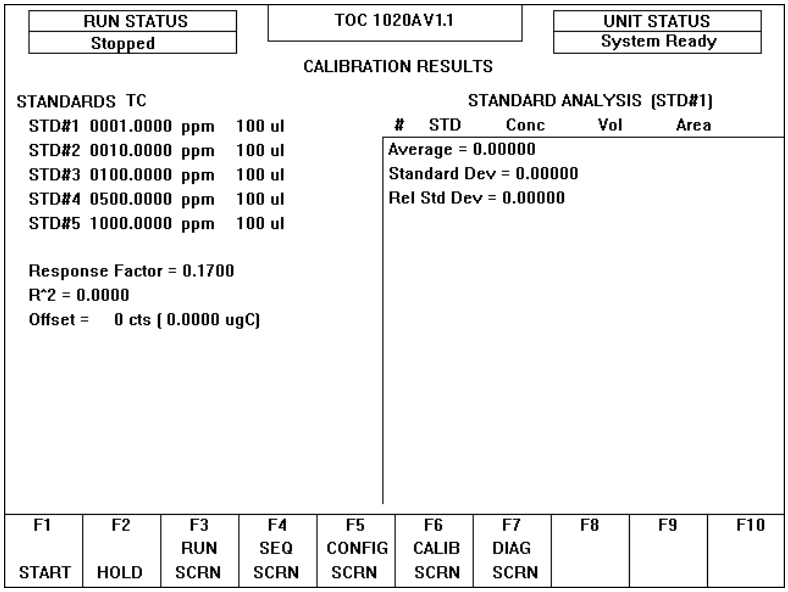
OUTLIER REMOVAL Allows the operator to select the relative standard deviation (**RSD**) and standard deviation (**Std. Dev.**) of the area for outlier recognition. One of these conditions must be met for the replicates to be accepted. If neither condition is met, additional reps will be analyzed to remove the outlier. The maximum number of additional reps is defined from the SEQUENCE SCREEN.

Date Displays the current date. This field may be edited for correction.

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 SCRN) 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.

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 (RF) Displays the response factor. The units are given in µg carbon per 1,000 area counts.

R^2 Displays the coefficient of correlation for the calibration line as it is being "built."

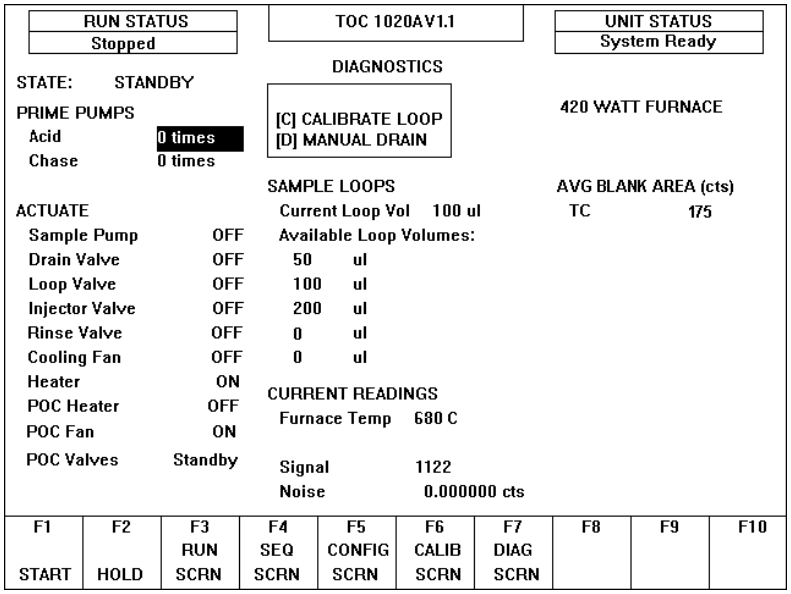
Offset Refers to the extrapolated y-intercept of the calibration curve. This value is calculated based on the calibration standards.

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 R^2 values. If all standards are deleted, the TOC Analyzer will use a default calibration Response Factor of 0.17 µgC/k counts.

DIAGNOSTICS SCREEN

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

STATE Displays the current mode of the TOC Analyzer.

PRIME PUMPS Allows the acid and chase 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 heater. 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 To aid 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 Vol Provides the same volume information as **Sample Vol** on the CONFIGURATION SCREEN and **STANDARDS** on the SEQUENCE SCREEN. Can be changed by scrolling [Page Up] or [Page Down] through the choices.

Available Loop Volumes Displays the sample loop volumes that are calibrated at the factory. If a sample loop needs to be calibrated, it is possible to calibrate the loop and enter the new volume on this screen.

Note: The loop volumes can be found on the sample loops that come with the TOC Analyzer and on the checkout sheet.

CURRENT READINGS Includes the furnace temperature and NDIR signal. The NDIR signal is displayed in area counts.

AVG BLANK AREA (cts) Displays the current TC blank value. The range is from 1–2,500 area counts.

Note: Values greater than 1,000 will elicite a warning message indicating possible decreased catalyst performance.

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 1020A 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 outlines the procedures for the operation of the Model 1020A TOC Analyzer using the control program ("Firmware" or "DOS 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 1020A Operator's Manual*.

For ease-of-operation, it is recommended that the TOC Analyzer remain ON with the carrier gas flowing at all times. If the TOC Analyzer is not used on a daily basis, it can be turned OFF and restarted as required; however, stabilization of the electronics and detector requires a 20- to 30-minute warm-up period each time the instrument is powered up.

Theory of Operation

The OI Analytical Model 1020A TOC Analyzer calculates organic carbon content from the difference between measured organic carbon and measured inorganic carbon. The instrument can be operated in TC, TIC, TOC, or POC mode. Before operation, it is important to develop both the TC and inorganic carbon (IC) calibration curves. Each measured analysis has a react and a detect time that is set to a default parameter for general analysis but can be optimized for specific analytical conditions. The TOC Analyzer will first perform replicate analyses of TC, then replicate analyses of TIC. The TOC concentration is calculated using the average of each value.

Equations 5.1–5.3 are used to calculate the mass and carbon concentrations in samples and check standards:

EQUATION 5.1

$$\textit{Sample Mass}\ (\mu gC) = (\textit{Area Counts} - \textit{TC Blank}) \times \left[\frac{RF}{1000}\right]$$

EQUATION 5.2

$$\textit{Check Standard Mass}\ (\mu gC) = (\textit{Area Counts} - \textit{Offset Area}) \times \left[\frac{RF}{1000}\right]$$

EQUATION 5.3

$$\textit{Carbon Concentration}\ (ppmC) = \left[\frac{\textit{Mass}\ (\mu gC) \times 1000}{\textit{Sample Volume}\ (\mu L)}\right]$$

Sample Introduction

Sample is introduced into the TOC Analyzer through a fixed volume sample loop to ensure reproducible replicates. The loop is filled either from the sipper tube or from the autosampler. Following sample introduction, the analysis for TC or TIC begins.

Total Carbon (TC) Analysis

TC analysis consists of a two-step process: TC Inject and TC Detect. During the TC Inject stage, the sample is transferred from the sample loop to the furnace, where all of the carbon is combusted to form carbon dioxide (CO_2). The flow upset generated by the expansion of water from liquid to gas phase is allowed to return to a constant flow rate by means of an expansion tube before the carbon dioxide is detected by the NDIR detector. The carbon dioxide is transferred to the detector through a permeation tube to remove all of the water from the sample. Carbon dioxide is then measured by the detector during the TC Detect stage and is vented to the atmosphere.

Total Inorganic Carbon (TIC) Analysis

TIC analysis consists of a two-step process: TIC Inject and TIC Detect. During the TIC Inject stage, the sample is transferred from the sample loop to the IC reaction chamber, where the sample is acidified with phosphoric acid (H_3PO_4). Acidification of the sample will shift the chemical equilibrium toward the production of carbon dioxide:

$$CO_3^{2-}(aq) + H^+ \leftrightarrow HCO_3^-(aq)$$

$$HCO_3^- + H^+ \leftrightarrow H_2O + CO_2\ (g)$$

During the TIC Detect stage, the inorganic carbon dioxide is purged from the sample and transferred to the NDIR detector, where it is detected and then vented to the atmosphere.

Total Organic Carbon (TOC) Analysis

TOC values are calculated by subtracting the average TIC value from the average TC value (see Equation 5.4).

EQUATION 5.4

$$TOC = TC - TIC$$

Purgeable Organic Carbon (POC) Analysis (Option)

The sample is transferred from the sample loop to the IC reaction chamber, where the sample is acidified with phosphoric acid (H_3PO_4). Acidification of the sample will release the inorganic carbon in the form of carbon dioxide (CO_2). The POC fraction of the sample and the inorganic carbon dioxide are transferred to the POC Module, where the POC fraction is collected on the trap. The inorganic carbon dioxide is not trapped and continues to the NDIR detector, where TIC is detected (see “Total Inorganic Carbon (TIC) Analysis” in this chapter). The trap is then heated, releasing the POC fraction. The POC is then transferred to the furnace, where all the carbon is combusted to carbon dioxide. This carbon dioxide fraction is transferred to the NDIR detector through a permeation tube to remove all of the water from the sample. Carbon dioxide is then measured by the detector during the POC Detect stage and is vented to the atmosphere.

Waste Drain

The system will drain the residual water from the IC chamber and perform any rinses mandated in the sequence.

Operational Parameters

The OI Analytical Model 1020A TOC Analyzer initiates using a default method of parameters for analysis at 680°C using a platinum catalyst in the combustion tube. These conditions are summarized in Table 5.1.

Table 5.1. Default Operational Parameters

Parameter	Default Setting
Sample Mode	TC
Standard Mode	TC
TC Inject	0:10
TC Detect	3:30
TIC Inject	0:10
TIC Detect	3:00
Sample ID	000
Sample Introduction	Sipper
Furnace Temperature	680°C
Sample Volume	100 µL
Acid Volume	200 µL
Rinse per Sample	OFF

The first step for the operation of the TOC Analyzer is the development of a calibration curve. The analyzer default settings are for a standard curve with TC standards of 10, 100, 500, and 1,000 ppm; however, other concentrations may be used by the operator.

Note: TC standards can be prepared using dried potassium hydrogen phthalate (KHP) in pure water. The default is analyzed in TC mode only because of the negligible content of inorganic carbon in KHP. Since the NDIR detector is selective to carbon dioxide, the introduction of carbon to the analyzer may be organic, inorganic, or a combination of the two. Organic KHP is recommended for ease-of-use.

Keyboard and Monitor Operation

The TOC Analyzer is controlled by keyboard and monitor using the MS-DOS-based firmware that is provided with the instrument. When using this configuration, all commands are entered as keystrokes. Table 5.2 lists the primary keys and their functions.

Table 5.2. 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

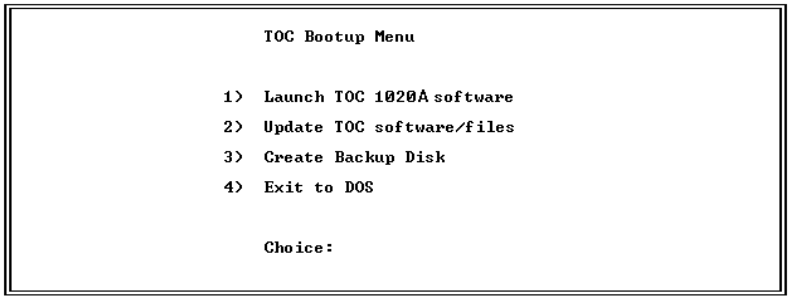
Starting Up the Unit

CAUTION:
Remove any floppy disks from the TOC Analyzer before operating the unit.

Powering up the TOC Analyzer loads the system program from the internal chip disk. This is only done for the initial start-up.

1. Turn on the power switch on the back panel of the instrument.

2. Turn on the power switch on the right side of the monitor. Following a system check, the **Bootup Screen** will appear:



3. Follow the installation instructions on the screen.

4. Press [1] to select **1) Launch TOC 1020A Software**.

Defining Sample Loop Size

Before operation, the sample loop size must be defined in the TOC firmware.

1. Press [F7] to access the DIAGNOSTICS SCREEN from the STARTUP SCREEN.

2. Using the arrow keys, position the cursor in the **Available Loop Volumes** field.

3. Enter the loop values previously recorded from the sample loops during installation (see “Installing the Sample Loops” in Chapter 3). Press [Enter] after each entry and use the Down [↓] arrow key to move to the next line.

   Example: For 53-µL, 91-µL, and 202-µL loops, type:

   “53” [Enter] [↓]
   “91” [Enter] [↓]
   “202” [Enter] [↓]

Calibrating the Autosampler

The autosampler carousel home position must be calibrated so that each vial is centered directly below the needle assembly. The carousel home position for the Model 1051 Autosampler must be set when:

- the Autosampler is being used for the first time;
- the TOC Program Disk is changed; or
- the data file is deleted from the TOC Program Disk.

Setting the home position will ensure that the needle consistently lines up with the center of each vial during programmed analyses and prevents needle damage and analysis errors.

Calibrate the Autosampler using the following procedure.

1. Verify that the TOC Analyzer and the Autosampler are not running.

2. Remove the carousel cover and place an open vial (without septum) in Position 1.

3. Replace the carousel cover.

4. Press [F5] to access the CONFIGURATION SCREEN.

5. Press [Alt][H] to rotate the carousel to the home position.

6. Center the needle above the vial by manually moving the platter under the carousel (see Figure 5.1).

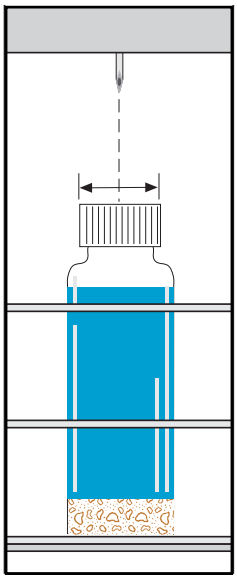


Figure 5.1. Needle Alignment

7. Press [Ctrl][Alt][C] to lock in the new position.

8. Press [Alt][N] to lower the needle and [Alt][O] to raise the needle to verify the alignment.

Calibrating the TOC Analyzer

A calibration curve must be developed. The analyzer default settings are for a standard curve with TC standards of 10, 100, 500, and 1,000 ppm; however, other concentrations can be used.

Note: TC standards can be prepared using dried potassium hydrogen phthalate (KHP) in pure water. The default is analyzed in TC mode only because of the negligible content of inorganic carbon in KHP. Since the NDIR detector is selective to carbon dioxide, the introduction of carbon to the analyzer may be organic, inorganic, or a combination of the two. Organic KHP is recommended for ease-of-use.

Setting Standard Concentrations

1. Press [F5] to access the CONFIGURATION SCREEN.

2. Use the [Page Up] or [Page Down] keys to set the **STD Mode** to TC, TIC, or POC.

3. Press [F4] to access the SEQUENCE SCREEN.

4. Press [M] **Modify STD** to modify standards information, if necessary. Use the Up [↑] or Down [↓] arrow keys to move the cursor within the standard information.

5. Press [Esc] to exit the **Standards** section of the SEQUENCE SCREEN. Changes will be saved automatically.

Running a Calibration Sequence

Separate TIC, TC, and POC calibration curves must be constructed prior to instrument operation. In the firmware operation, TC, TIC, and POC calibration standards may **not** be run in the same sequence. To run a calibration sequence for TC standards, first set the standard mode on the CONFIGURATION SCREEN to TC and then continue with the proceeding steps. Following this, reset the standard mode to TIC and rerun the sequence.

1. Press [F4] to access the SEQUENCE SCREEN.

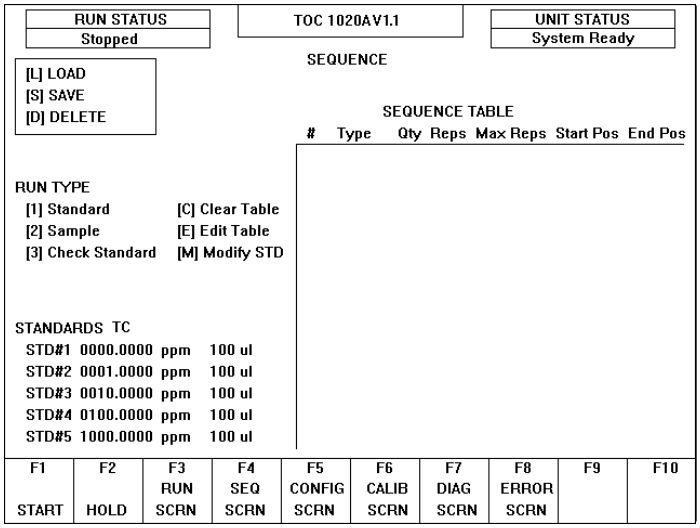
2. Press [1] to select **Standard**.

3. Enter the desired 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 standard and press [Enter].

5. Repeat steps 2–4 until all standards to be run are entered into the sequence.

Note: Press [Esc] to clear the line currently being entered in the **Sequence Table**.



6. Press [F1] to start the calibration sequence.

Note: To hold the run, press [F2]. The run can then be resumed by pressing [F1] or aborted by pressing [F2] again. The instrument processing will pause; however, chemical processes will not stop.

Note: While the TOC Analyzer is not running, calibration sequences, as well as other sequences, can be saved from the SEQUENCE SCREEN by pressing [S] **SAVE** after programming the sequence.

Note: The response factor for an acceptable calibration should be in the range of 0.13–0.21 µgC/k counts.

Note: Only ten replicates are used in the calculation of the calibration.

Running Check Standards

Running check standards allows the operator to run a known standard to verify the calibration of the TOC Analyzer without affecting the calibration. Check standards values will have the calibration offset value subtracted. Check standards are recalled from the same standards as programmed on the SEQUENCE SCREEN. It is important that the mode of standard on the SEQUENCE SCREEN matches the mode on the CONFIGURATION SCREEN (e.g., a check standard of TC in the sequence should have the CONFIGURATION SCREEN set to TC STD mode). For low-level samples, use reagent water for the check standard.

1. Press [F4] to access the SEQUENCE SCREEN.

2. Press [3] 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 (1–10) to be run of that particular check standard and press [Enter].

5. Repeat steps 2–4 until all check standards to be run are entered into the sequence.

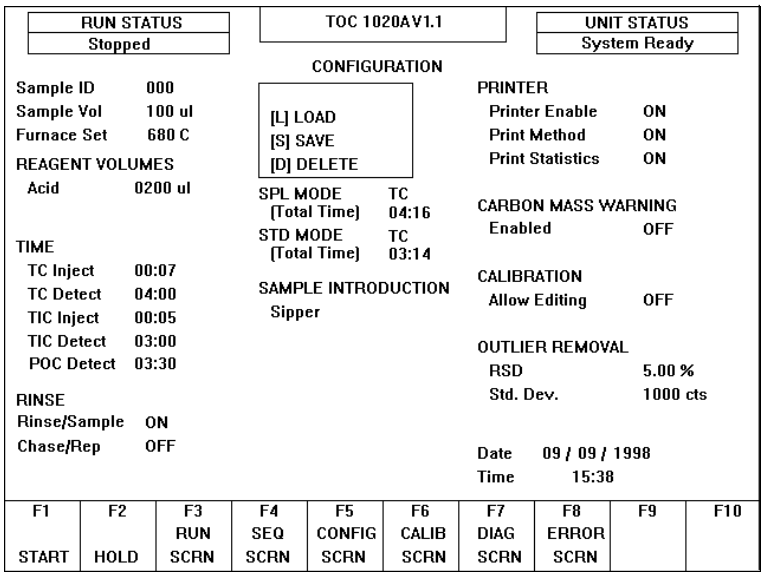
6. Press [F1] to start the check standard sequence.

Note: While the TOC Analyzer 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).

Configuring the TOC Analyzer

The CONFIGURATION SCREEN provides access to the instrument parameters, which can then be saved as methods. The default parameters (Table 5.1) can be optimized for specific analyses.

1. Press [F5] to access the CONFIGURATION SCREEN



2. Specify the **Sample ID**. The following numbers will advance incrementally.

3. Select the **Sample Volume** using the [Page Up] and [Page Down] keys. Loop volumes must be defined in the DIAGNOSTICS SCREEN. The default is 100 µL.

4. Verify that the furnace temperature, under **Furnace Set**, is set properly for the current analysis. The default is 680°C for operation with the platinum catalyst bed. Do not exceed 720°C for platinum catalytic operation.

5. Set the **Reagent Volume**, depending on the amount of inorganic carbon (IC) present in the sample. Settings are available in 100-µL increments.

6. Enter the **Time** settings for the current analysis. These settings are optimized for aqueous samples (see Table 5.1 for operational parameters). Samples with higher viscosity (e.g., salts and brines) may require longer inject and detect times and should be varied accordingly.

7. Set the **Rinse/Sample** to configure the TOC Analyzer to run a rinse per sample. **Rinse/Sample** is defaulted to "OFF." Set the **Chase/Rep** to configure the TOC Analyzer to run a chase between reps. **Chase/Rep** is defaulted to "OFF."

8. Select the **Sample (SPL) Mode** by using the [Page Up] and [Page Down] keys. The default setting is TC. During the final check-out procedure, KHP is used as an organic standard. Since this standard contains only TOC with no IC, the TOC Analyzer is operated in the TC mode only. The TC values reported are also the organic values. It is often convenient to operate the instrument in TC standards mode to reduce analysis time and operate in TC/TIC/TOC sample mode. Operation of standards and samples in different modes does not affect the analysis, as the NDIR detector is detecting total carbon dioxide in both cases.

CAUTION:
Data is not saved in the TOC firmware and must be printed to retain a record.

9. Enable or disable the print options as necessary.

10. To modify the current calibration curve, under **Calibration** turn **Allow Editing** ON.

11. Press [S] to save the modified configuration.

Creating and Running a Sequence

Sequencing allows the operator to program and run combinations of samples, standards, and check standards.

1. Press [F4] to access the SEQUENCE SCREEN.

2. Press the appropriate key ([1], [2], or [3]) to select **Standard**, **Sample**, or **Check Standard**, respectively.

3. If **Standard** is selected, the standard concentration can be edited by pressing [M] (**Modify STD**).

4. Enter the number of samples to be analyzed and press [Enter].

5. Enter the number of replicates to be run of each sample and press [Enter].

6. Repeat step 2–4 until the sequence is programmed (see “Example” in this chapter).

7. Repeat until the sequence is programmed.

8. 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 entire table.

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

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

Example

The following is an example of the steps used to prepare a four-point calibration sequence of 1, 10, 50, and 100 ppm and then to run five samples in triplicate, a 50-ppm check standard in duplicate, and three more samples in triplicate.

1. Press [F4] to access the SEQUENCE SCREEN.

2. Enter the 1.00 ppm standard to be run in triplicate as Standard 1.
   [1] [Enter] [3] [Enter] [Enter]

3. Enter the 10.00 ppm standard to be run in triplicate as Standard 2.
   [1] [2] [Enter] [3] [Enter] [Enter]

4. Create the 50.00 ppm standard and enter it to be run in triplicate as Standard 5.
   [M] [↑] [5] [0] [Enter] [Esc]
   [1] [5] [Enter] [3] [Enter] [Enter]

5. Enter the 100.00 ppm standard to be run in triplicate as Standard 3.
   [1] [3] [Enter] [3] [Enter] [Enter]

6. Enter the five samples to be run in triplicate.
   [2] [5] [Enter] [3] [Enter] [Enter]

7. Enter the 50 ppm check standard to be run in duplicate as Standard 5.
   [3] [5] [Enter] [2] [Enter] [Enter]

8. Enter the final three samples to be run in triplicate.
   [2] [3] [3] [Enter] [Enter]

9. Name the sequence “Samples” and save the sequence.
   [S] [↑] [s] [a] [m] [p] [l] [e] [s] [Enter]

10. Press [F3] to access the RUN SCREEN.

11. Place the sipper tube into the 1-ppm standard and press [F1].

Note: To pause the analysis, press [F2]. Resume the run by pressing [F1]. To stop the analysis, press [F2] twice.

Determining a TC Blank Value

The following procedure is used to establish a TC blank value. It is recommended that a new blank value be determined each time the instrument is calibrated. In order to obtain the most accurate and representative value of the background carbon level, the use of ASTM Type I water or its equivalent is recommended.

1. Check the sample loop size used and ensure that it is the size that will subsequently be used in calibration and sample analysis.

2. Press [F5] to access the CONFIGURATION SCREEN. Set **Sample Introduction** to **Sipper.**

3. Fill a clean vessel with reagent water and cover with Parafilm® to prevent absorption of carbon dioxide from the atmosphere.

4. Press [F4] to access the SEQUENCE SCREEN.

5. Press [2] to select **Sample**.

6. Enter [1] for **Qty** and [10] for **Reps**.

7. Press [F1] to start the analysis.

8. At the end of the sequence, determine if the blank area counts have stabilized. A difference of <40–50 area counts is usually sufficient. If the baseline is not stable, repeat steps 4–7.

9. When the area counts have stabilized, average the last 3–5 values to obtain the TC blank value.

10. Press [F7] to access the DIAGNOSTICS SCREEN.

11. Enter the TC blank value calculated in step 9 as the **TC Blank**.

Shutting Down the Unit

The following procedure should be followed to allow the components to properly cool and to minimize contamination.

1. Press [F2] twice to end all operations on the TOC Analyzer.
2. Turn off the power switch on the right back panel of the instrument.
3. Allow the instrument to cool for 10 minutes with the carrier gas flowing.
4. Remove and empty the humidifier vessel. Reattach the humidifier vessel.
5. Turn off the carrier gas supply.

Notes

Chapter 6
Maintenance

This chapter discusses both the scheduled and nonscheduled maintenance of the Model 1020A Combustion TOC Analyzer, starting with some general information and a maintenance schedule.

Scheduled Maintenance

It is recommended that the operator set up an instrument logbook to record instrument operation time and document periodic maintenance. This logbook can be used to record the results of inspections and component replacements necessary for proper maintenance of the TOC Analyzer.

For the most reliable performance of the TOC Analyzer, the schedule of routine maintenance shown in Table 6.1 should be followed. (Scheduled hours refer to number of hours of operation.)

Table 6.1. Schedule for Routine Maintenance

Maintenance Item	Schedule
Acid reagent bottle	As needed
Rinse bottle	As needed
Catalyst	As needed
Humidifier water	As needed
NDIR zero	100 hours
Sample pump tubing	2,000 hours
Permeation tube	12 months
Halide scrubber	4 months or as needed

For ease-of-maintenance, the plumbing tubing is color-coded. See Table 6.2.

Table 6.2. Color Codes for Tubing

Tubing Color	Use
Black	Waste
Green	Inject
Blue	Sample
Yellow	CO_2
Clear	Gas
Red	Acid

Acid Reagent Bottle

The volume of acid reagent in the acid bottle on the side of the TOC Analyzer should be monitored periodically according to the number of analyses and volume of reagent used per sample. Reagent should be added to keep the bottle from being completely emptied. Operating the reagent pump without liquid is not recommended. See Chapter 1, “Introduction,” for preparing the reagent to add to this bottle.

Rinse Bottle

The volume of the rinse reservoir on the side of the TOC Analyzer should be periodically monitored according to the number of analyses. Maintain reagent water for system rinses. It is recommended that the rinse bottle be cleaned approximately every two weeks to avoid bacterial/algal growth.

Catalyst Conditioning

It is recommended that the catalyst be conditioned before analysis when installing a new catalyst, changing the catalyst, when the unit has not been operated for a long period of time, or the integrated area counts increase or become variable.

Note: These command descriptions are for operating the analyzer using a keyboard and monitor. For WinTOC operation, refer to the *WinTOC 1020A Operator’s Manual*.

1. Verify that the flow is at 150 ±10 mL/min at the NDIR vent.
2. Remove the left bay cover.
3. Install the 100-µL sample loop and replace the left bay cover.
4. Press [F5] for the CONFIGURATION SCREEN.
5. Turn **Chase/Rep** ON.
6. Enter the actual sample loop volume under **Sample Vol**.
7. Set the furnace temperature to 680°C.
8. Place the sample inlet in a bottle of reagent water.
9. Press [F4] for the SEQUENCE SCREEN.
10. Press [2] to select **Sample**.
11. Enter [1] for **Qty** and [15] for **Reps**.
11. Press [F1] to start the analysis.

12. Observe the injection through the hole in the top of the furnace cover (see "Injector Cap Operation" in this chapter). Verify that the injection stream and/or droplets are centered in the furnace and are not spraying toward the sides of the combustion tube, and that the injection occurs within three seconds.

13. Correct the alignment. If necessary, see "Injector Cap Operation" in this chapter.

14. If the injection time is longer than three seconds, verify the flow rate and check for obstructions.

15. Once the inject time and alignment are verified to be correct, continue running the 15 replicates of reagent water.

16. The carbon dioxide peak produced from the injections should decrease steadily over the first 20 injections and stabilize at <1,000 counts. If peak areas are >1,000 counts after 20 injections, refer to "Cleaning the Catalyst" in this chapter.

17. Replace the left bay cover.

Cleaning the Catalyst

1. Turn off the power switch on the right back panel of the instrument.

2. Allow the instrument to cool for approximately 30 minutes with the carrier gas flowing. Accelerate cooling by removing the left cover from the instrument and removing the heat shield.

3. Once the furnace is cooled sufficiently, slide the spring clamp from the end of the Viton tubing.

4. Remove the Viton tubing from the end of the combustion furnace. It may be necessary to cut the tubing.

5. Remove the fitting from the injector cap. Unscrew the injector cap from the 18-mm knurled nut.

6. Once all of the connections have been removed from the combustion furnace, lift the combustion tube from the combustion furnace.

7. Empty the contents of the combustion tube into a clean 250-mL beaker. It may be necessary to fill the combustion tube with reagent water and soak the contents for 1–2 minutes.

8. Remove and discard the glass wool. Leave the platinum gauze and quartz chips in the beaker to be cleaned along with the catalyst.

9. Add approximately 100 mL of 5% hydrochloric acid (HCl) to the beaker with the catalyst. Stir thoroughly. Allow catalyst to soak for approximately 5–10 minutes, stirring frequently.

10. Decant the HCl solution and rinse the catalyst thoroughly with several aliquots of 100–150 mL of reagent water. Check the pH of the supernatant. When the pH is neutral, the catalyst has been sufficiently rinsed.

11. Dry the catalyst in a 100°C oven for approximately ½–1 hour.

12. Allow the contents to cool. Remove the platinum gauze and the quartz chips from the catalyst beads.

13. Repack the combustion tube. See "Replacing/Packing the Combustion Tube" in this chapter.

Checking and Adding Water to the Humidifier Vessel

The TOC Analyzer has a humidifier (humidifier vessel) to reduce stress on the catalyst (see Figure 2.1). The vessel should be visually checked to ensure the proper level of reagent water. The humidifier vessel should be between ⅓ and ¾ full of reagent water.

1. Locate the humidifier vessel on the front of the TOC Analyzer (Figure 2.1).

2. Check the water level in the humidifier vessel and ensure that the humidifier vessel is between ⅓ and ¾ full.

3. If the water level is low, remove the humidifier vessel from the clip on the front panel of the TOC Analyzer.

4. Unscrew and remove the cap from the humidifier vessel.

5. Add reagent water to the vessel until it is ¾ full.

6. Replace the cap and screw it onto the humidifier vessel.

7. Replace the humidifier vessel on the front panel.

Gas Service

Gas consumption is listed in Chapter 1, "Introduction." Standard 2,000-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 periodically to confirm sufficient gas for planned operations.

Injector Cap Operation

The injector cap should be inspected periodically to ensure that it is properly injecting the sample into the combustion tube. If corrosive or seawater samples are analyzed routinely, this inspection should be performed every 400 hours. To inspect the injector cap:

1. Remove the left bay cover.
2. While the TOC Analyzer is performing a TC injection, view the injector cap through the view port of the furnace heat shield.
3. The injection should appear as a stream from the injector cap.

Note: If this stream is inconsistent or nonexistent, the injector cap needs to be cleaned. See "Cleaning the Injector Cap" in this chapter.

4. Replace the left bay cover.

NDIR Zero

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.*

The NDIR detector zero (baseline) will fluctuate up or down during periods of nonuse. This is due to factors such as operating temperature, how long the NDIR purge has been on expelling ambient carbon dioxide, or purity of gases. However, under routine operating conditions, the baseline reading should be set between 4,000 and 8,000 for optimum range and linearity response. This adjustment should be checked after every 100 hours of operation (corresponding to gas service maintenance).

Note: These command descriptions are for operating the analyzer using a keyboard and monitor. For WinTOC operation, refer to the *WinTOC Model 1020A Operator's Manual.*

To adjust the NDIR zero:

1. Press [F3] for the RUN SCREEN.

CAUTION: *Do not make adjustments to the GAIN potentiometer as this will affect the NDIR detector linearity.*

2. Verify that the TOC Analyzer is in the Standby State.
3. Slowly turn the NDIR Zero OFFSET adjustment (on the NDIR enclosure) (see Figure 6.1) clockwise to increase the baseline (positive shift) or counter-clockwise to decrease the baseline (negative shift). Set the output between 4,000 and 8,000.
4. Allow the TOC Analyzer to perform several automated analyses and recheck the baseline with the TOC Analyzer in the Standby State. Make any necessary adjustments as described in step 3.

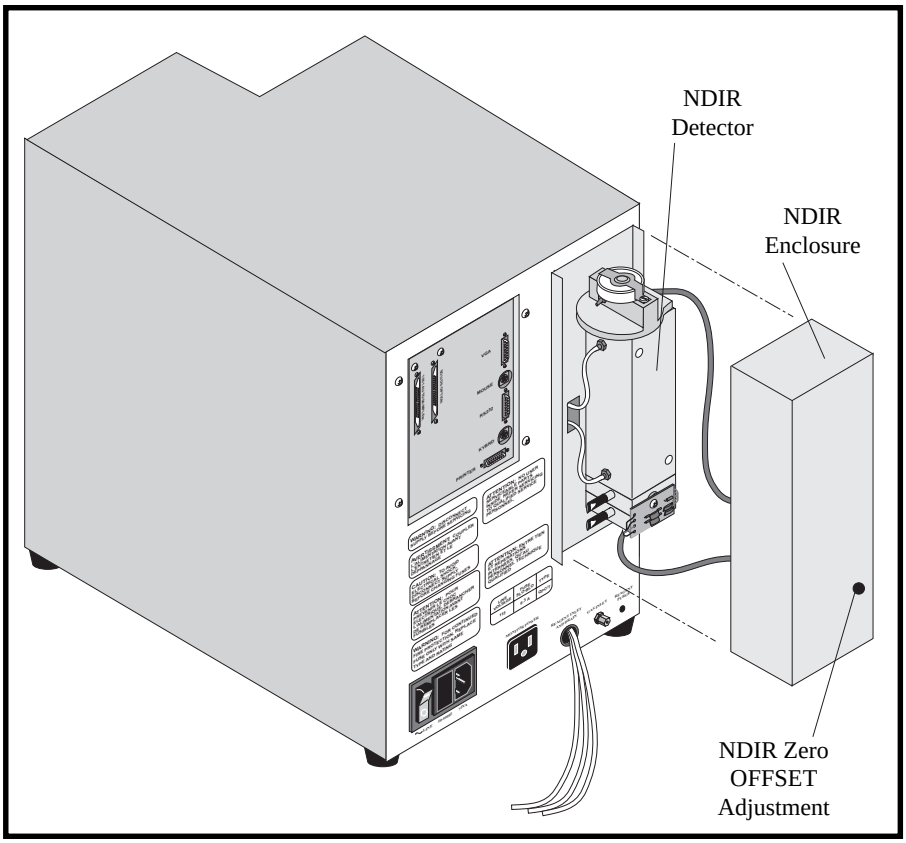


Figure 6.1. Nondispersive Infrared (NDIR) Detector

Sample Pump

Note: These command descriptions are for operating the analyzer using a keyboard and monitor. For WinTOC operation, refer to the *WinTOC 1020A Operator's Manual*.

This procedure applies to the peristaltic sample pump mounted inside the left bay (see Figure 6.2). It is used to aspirate samples through the loop sampling inlet and the sample loop. The sample 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 2,000 hours of operation. More frequent inspections may be necessary if samples containing strong acids, solvents, or bases are run.

1. Remove the left bay cover.

2. Remove the plastic barb fittings from both ends of the pump tube.

3. With a small flat-blade screwdriver, carefully pry apart the teeth of the plastic retaining clamp on the inlet leg of the neoprene tube and remove the retaining clamp from the end of the blue tubing.

4. Press [F7] for the DIAGNOSTICS SCREEN.

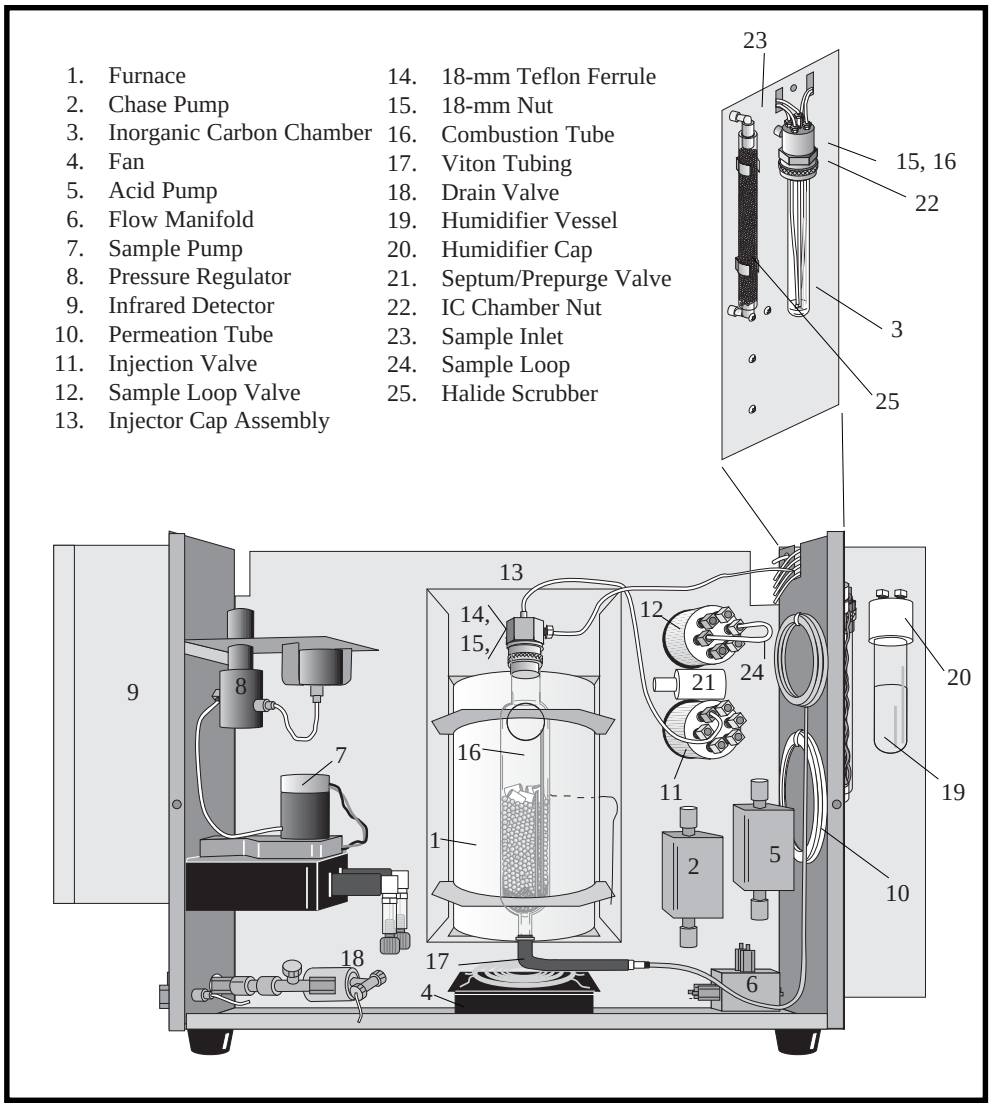


Figure 6.2. Left Interior of the Model 1020A TOC Analyzer

5. Under the **ACTUATE** section, turn on the **Sample Pump**.

6. While the pump is turning, pull the sample outlet leg to remove the tubing from the pump housing.

7. Inspect the tubing for excessive wear, holes or cracks, and replace it if signs of these 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. Installation of the tubing into the pump housing is the reverse of steps 1–6.

8. Replace the left bay cover.

Permeation Tube

A gas permeation tube is plumbed between the effluent of the IC chamber and the NDIR detector. The gas permeation tube is a coaxial tube set containing a hydrophilic membrane designed to selectively remove water vapor from mixed gas streams. 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 diffuse across the walls of the tubing.

The ion membrane is chemically resistant to most gases and liquids; however, the drying capacity may be decreased if the membrane becomes contaminated with nonvolatile liquids or salts. The permeation tube should be replaced annually as preventive maintenance.

NDIR Linearization Check

All nondispersive infrared detectors produce a nonlinear response unless electronically corrected by a linearizer board, or in the case of the TOC Analyzer, the output response is corrected algebraically.

The detector in the TOC Analyzer has been linearized over a range of 0–250 µgC and should remain linearized indefinitely. However, quality assurance practices and proper maintenance procedures should include routine linearity checks.

If a linearity problem is suspected, contact the OI Analytical Technical Support Department at (800) 336-1911 or (979) 690-1711 for assistance.

Nonscheduled Maintenance

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

Calibrating the Acid Pump

Note: These command descriptions are for operating the analyzer using a keyboard and monitor. For WinTOC operation, refer to the *WinTOC 1020A Operator's Manual*.

This calibration applies to the acid pump, which can be identified by the red fittings (see Figure 6.2).

1. Remove the left bay cover.

2. Loosen the 18-mm nut on the IC chamber, and remove the IC chamber glassware.

3. Press [F7] for the DIAGNOSTICS SCREEN.

4. Under the **PRIME PUMPS** section, program **Acid** for 10 times.

5. Place the end of the ⅛" red acid line tubing into a measuring vessel and press [Enter].

6. When the pump stops pumping, use a 2-mL syringe to measure the volume of the contents in the vessel.

7. To adjust the pump volume, loosen the ½" locknut on the threaded shaft at the bottom of the pump.

8. If the volume of the vessel is more than 1 mL, turn the ¼" shaft (using a ¼" wrench) located below the pump clockwise. If the volume of the vessel is less than 1 mL, turn the shaft counterclockwise.

9. Repeat steps 4–8 until the volume dispensed is between 0.95 mL and 1.05 mL.

10. Tighten the locknut and check the volume again.

11. Reinstall the IC chamber glassware and tighten the 18-mm nut.

12. Replace the left bay cover.

Calibrating the Chase Pump

Note: These command descriptions are for operating the analyzer using a keyboard and monitor. For WinTOC operation, refer to the *WinTOC 1020A Operator's Manual*.

This calibration applies to the acid pump, which can be identified by the tan PEEK® fittings (see Figure 6.2).

1. Remove the left bay cover.

2. Loosen the 18-mm nut on the IC chamber, and remove the IC chamber glassware.

3. Verify that the water bottle is filled.

3. Press [F7] for the DIAGNOSTICS SCREEN.

4. Under the **PRIME PUMPS** section, program **Chase** for 10 times.

5. Place the end of the ⅛" green chase line tubing into a measuring vessel and press [Enter].

6. When the pump stops pumping, use a 2-mL syringe to measure the volume of the contents in the vessel.

7. To adjust the pump volume, loosen the ½" locknut on the threaded shaft at the bottom of the pump.

8. If the volume of the vessel is more than 0.5 mL, turn the ¼" shaft (using a ¼" wrench) located below the pump clockwise. If the volume of the vessel is less than 0.5 mL, turn the shaft counterclockwise.

9. Repeat steps 4–8 until the volume dispensed is between 0.45 mL and 0.55 mL.

10. Tighten the locknut and check the volume again.

11. Reinstall the IC chamber glassware and tighten the 18-mm nut.

12. Replace the left bay cover.

Performing a System Leak Check

1. Remove the left bay cover.

2. Using the pressure regulator inside the TOC Analyzer, set the internal system pressure to 12 psi.

3. Locate the NDIR vent line on the NDIR assembly (see Figure 6.3).

4. Plug the line with a suitable plug.

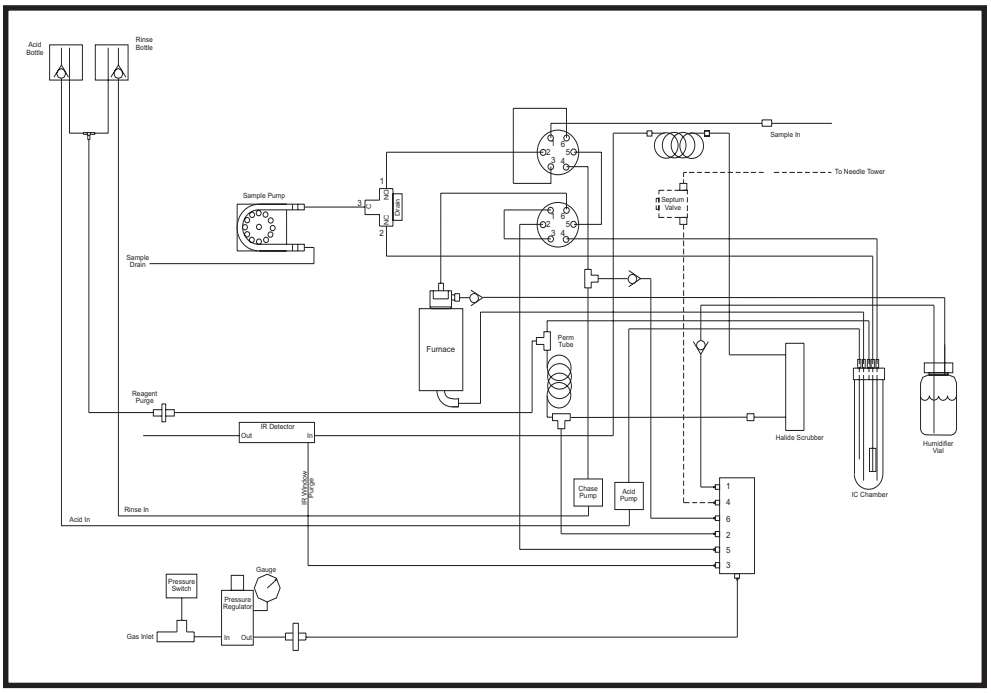


Figure 6.3. Model 1020A TOC Analyzer Flow Diagram

5. Plug the purge lines to acid/water.

6. Observe the humidifier. Bubbles should decrease and stop within 10 minutes.

7. Remove the plugs.

8. Reset the system pressure to approximately 18 psi, and readjust the IR flow to 150 ±10 mL/min.

9. Replace the left bay cover.

Note: If there is a leak, the 18-mm Teflon ferrule on the top of the furnace is a good place to start.

Replacing/Packing the Combustion Tube

WARNING: *The injector cap assembly and combustion tube may be hot! Make sure that the furnace has cooled before removing the heat shield*

For 900°C Operation

1. Turn off the TOC Analyzer and allow the furnace to cool.
2. Remove the left bay cover.
3. Loosen the four screws that hold the furnace heat shield in place. Remove the shield by lifting up and sliding down and out.
4. Loosen the 18-mm knurled nut located on the top of the combustion tube. Remove the injection cap assembly.
5. Gently lift the combustion tube out of the furnace.
6. Pour out the old quartz and zirconia, and clean the combustion tube with reagent water.
7. If the platinum gauze is not already in place, fit the platinum gauze (Part #281121) on the bottom of the combustion tube (Figure 6.4A).
8. Insert the fill tube into the combustion tube. Using the funnel, pour approximately 2 cm of zirconia into the fill tube (Figure 6.4A).
9. Lift the fill tube slowly and allow the zirconia to fill the combustion tube (Figure 6.4B). Continue to add zirconia into the combustion tube until it reaches a level of 10.25 cm (4") from the top of the combustion tube (Figure 6.4C).

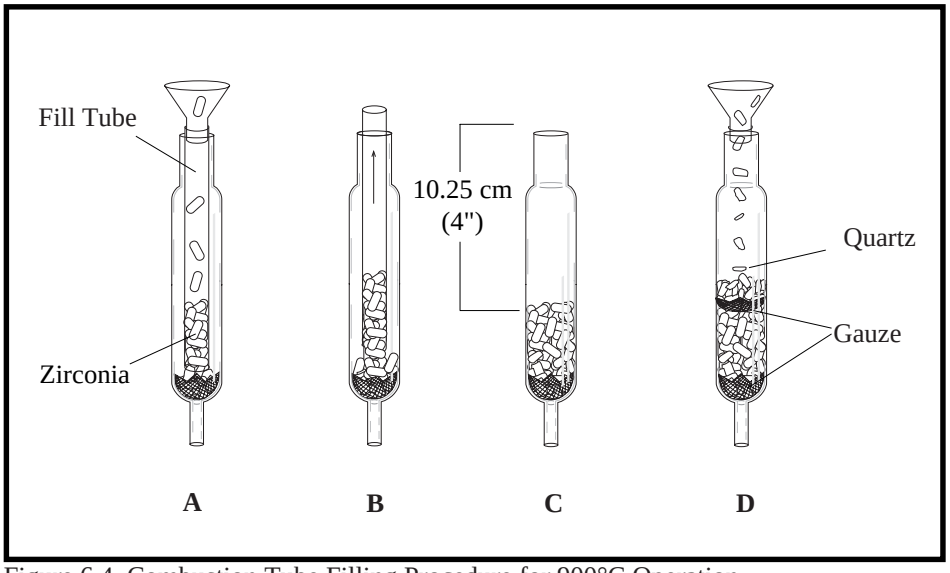


Figure 6.4. Combustion Tube Filling Procedure for 900°C Operation

10. Place a piece of platinum gauze mesh on top of the zirconia.

11. Using the funnel, pour 1.0 cm (0.4") of quartz on top of the platinum gauze.

12. Verify that both gauze are in place on the finished combustion tube (Figure 6.4D).

13. Gently insert the filled combustion tube into the top of the furnace.

14. Attach the 18-mm nut and the 18-mm Teflon ferrule to the top of the combustion furnace, facing up, and attach the injector assembly. Tighten the 18-mm knurled nut until tight.

15. Replace the combustion furnace heat shield and left bay cover.

16. Set the furnace temperature to 900°C and flush the system by running reagent water samples until the baseline stabilizes.

WARNING: *The injector cap assembly and combustion tube may be hot! Make sure that the furnace has cooled before removing the heat shield*

For 680°C Operation

1. Turn off the TOC Analyzer and allow the furnace to cool.

2. Remove the left bay cover.

3. Loosen the four screws holding the furnace heat shield in place. Remove the shield by lifting up and sliding down and out.

4. Loosen the 18-mm knurled nut located on the top of the combustion tube. Remove the injection assembly.

5. Gently lift the combustion tube out of the furnace.

6. Pour out the old catalyst and clean the combustion tube with reagent water.

7. If the platinum gauze is not already in place, fit the platinum gauze (Part #281121) on the bottom of the combustion tube (Figure 6.5A).

8. Insert 0.5" (1.27 cm) of quartz wool (Part #144501) on top of the mesh.

9. Insert the fill tube. Using the funnel, pour approximately 2 cm of platinum catalyst into the fill tube (Figure 6.5A).

10. Lift the fill tube slowly, allowing the catalyst to fill the combustion tube (Figure 6.5B). Continue to pour catalyst into the combustion tube until it reaches a level 11 cm from the top of the tube (Figure 6.5C).

11. Using the funnel, pour approximately 1 cm of quartz on top of the catalyst bed (Figure 6.5D).

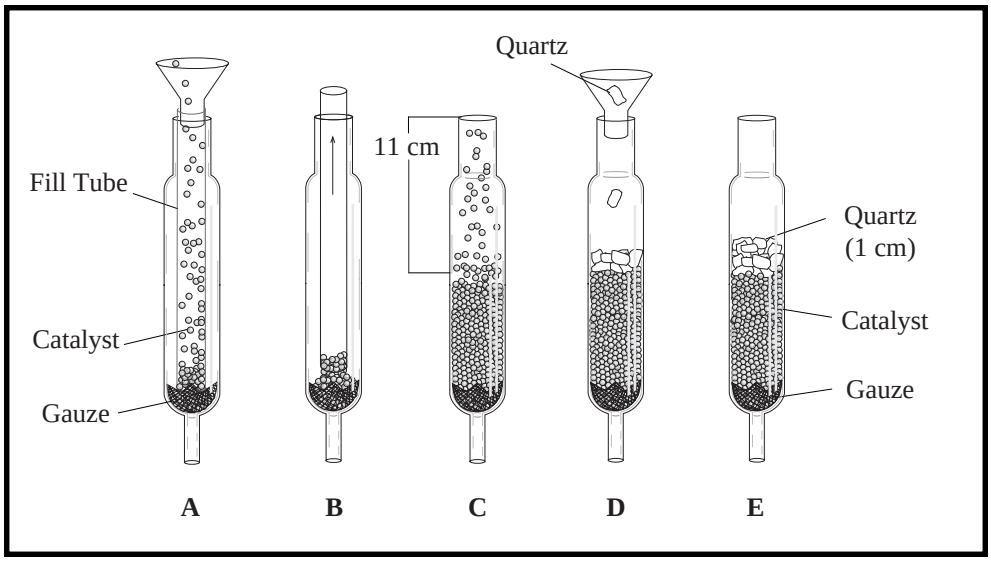


Figure 6.5. Combustion Tube Filling Procedure for 680°C Operation

12. Gently insert the filled combustion tube into the top of the furnace.

13. Attach the 18-mm knurled nut and the 18-mm Teflon ferrule to the top of the combustion tube, facing up, and attach to the injector assembly. Tighten the 18-mm knurled nut until tight.

14. Replace the combustion furnace heat shield and the left bay cover.

15. Set the furnace temperature to 680°C and flush the system by running reagent water samples until the baseline stabilizes.

Cleaning the Injector Cap Assembly

WARNING: *The injector cap assembly and combustion tube may be hot! Make sure that the furnace has cooled before removing the heat shield*

1. Turn off the TOC Analyzer and allow the furnace to cool.

2. Remove the left bay cover.

3. Loosen the four screws holding the furnace heat shield in place. Remove the shield by lifting up and sliding down and out.

4. Disconnect the green sample transfer tubing from the top of the injector cap assembly. See Figure 6.6.

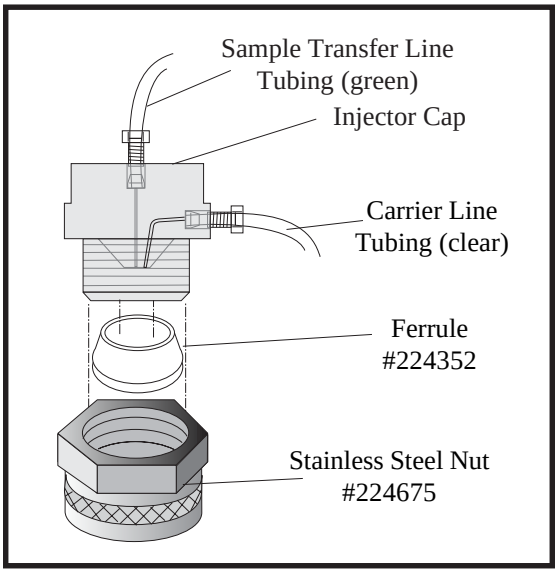


Figure 6.6. Injector Cap Assembly

5. Remove the ⅛" clear TFE carrier line tubing from the side of the injector cap assembly.

6. Using the two wrenches provided, loosen the stainless steel nut (Part #224675) and remove the injector cap assembly.

7. Remove the stainless steel nut from the injector cap and discard the ferrule.

8. Using a soft cloth to remove debris, rinse the injector cap with reagent water. A Scotch-Brite® pad may be used to remove stains or hard residue. To prevent damage or burrs that cause injection stream disturbances, use caution around the sample outlet.

9. Carefully insert a properly sized cleaning wire through the tubing inlets. If the obstructions cannot be easily removed with the cleaning wire, the injector cap assembly must be replaced.

10. Install a new ferrule (Part #224352) into the stainless steel nut. Reattach the injector cap to the nut and ferrule.

11. Install the injector cap assembly onto the combustion tube. Ensure that the cap is fully seated. Use the two 18-mm wrenches to firmly tighten the stainless steel nut.

12. Install the sample transfer tubing and carrier line tubing into the injector cap assembly.

13. Tighten the sample and carrier line nuts approximately ⅛-turn past fingertight. Excessive tightening will constrict the TFE tubing and will result in poor flow performance.

14. Recheck the total flow from the vent outlet on the back of the TOC Analyzer to verify that the injector cap assembly is seated properly.

15. Inspect the injector cap operation as described in “Injector Cap Operation” in this chapter

16. Replace the combustion furnace heat shield and left bay cover.

Replacing the Injector Cap Assembly

WARNING: *The injector cap assembly and combustion tube may be hot! Make sure that the furnace has cooled before removing the heat shield*

1. Turn off the TOC Analyzer and allow the furnace to cool.

2. Remove the left bay cover.

3. Loosen the four screws holding the furnace heat shield in place. Remove the shield by lifting up and sliding down and out.

4. Disconnect the green sample transfer tubing from the top of the injector cap assembly. See Figure 6.6.

5. Remove the ⅛" clear TFE carrier line tubing from the side of the injector cap assembly.

6. Using the two 18-mm wrenches provided, loosen the stainless steel nut (Part #224675) and remove the injector cap assembly.

7. Install the new injector cap assembly onto the combustion tube. Ensure that the cap is fully seated. Use the two 18-mm wrenches to firmly tighten the stainless steel nut.

8. Install the sample transfer tubing and the carrier line tubing into the injector cap assembly.

9. Tighten the sample and carrier line nuts approximately ⅛-turn past fingertight. Excessive tightening will constrict the TFE tubing and will result in poor flow performance.

10. Recheck the total flow from the vent outlet on the back of the TOC Analyzer to verify that the injector cap assembly is seated properly.

11. Inspect the injector cap operation as described in "Injector Cap Operation" in this chapter

12. Replace the combustion furnace heat shield and left bay cover.

Changing Sample Loops

The TOC Analyzer contains a sample loop valve with a sample loop attached. To convert to other loop sizes, the current loop is removed and another loop is attached to the valve.

Note: These command descriptions are for operating the analyzer using a keyboard and monitor. For WinTOC operation, refer to the *WinTOC 1020A Operator's Manual*.

1. Remove the left bay cover.

2. Locate the blue sample loop on the sample loop valve. See Figure 6.2.

3. Remove the sample loop by unscrewing the fittings on the loop.

4. Install the new sample loop by screwing the fittings on the loop into the sample loop valve.

5. Replace the left bay cover.

6. Press [F5] for the CONFIGURATION SCREEN and change the **Loop Size**.

Flow Adjustment

1. If using Firmware, press [F3] to verify that the TOC Analyzer is in the Standby State. If using WinTOC, go to the Status Screen and verify that the TOC Analyzer is in the Standby State.

2. Remove the left bay cover.

3. Locate the NDIR vent line.

4. Connect a flowmeter to the NDIR vent line.

5. Measure the flow. It should be 150 ±10 mL/min.

6. If this is not the case, adjust the regulator until the proper flow is reached.

7. Verify that the pressure regulator gauge reads 18 psi.

8. Remove the flowmeter.

9. Replace the left bay cover.

Calibrating Sample Loops

Note: These command descriptions are for operating the analyzer using a keyboard and monitor. For WinTOC operation, refer to the *WinTOC 1020A Operator's Manual*.

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. The program will fill the loop five times, so divide the measured amount by five to arrive at the loop volume.

6. Once the specification for the loop is met, enter the **Current Loop Vol** under the **SAMPLE LOOPS** section on the DIAGNOSTICS SCREEN.

Chapter 7
Troubleshooting

The following is a list of Model 1020A TOC Analyzer 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 Component Symptoms

Symptom	Probable Cause	Corrective Action
Sample does not properly aspirate into the Model 1020A from the sample bottle or autosampler	Worn sample pump tubing	Replace the sample pump tubing; see “Sample Pump” in Chapter 6
	Leak in sampling line	Verify that the fittings on the tubing from the loop injection port to the sample pump are tight
	Sample loop not properly tightened	Check sample loop connections for finger-tightness
	Sample pump tubing pinched shut from lack of use	Service or replace sample pump tubing; see Chapter 6, “Maintenance”
Peaks have shoulder-like tails	Poor catalyst	Condition catalyst; see “Catalyst Conditioning” in Chapter 6
	Poor injection	Inspect the injector tubing and injector cap
		Verify injection flow
“W” shaped or negative inflection on carbon dioxide peak (TIC or TC)	Restriction in system	Condition catalyst; see “Catalyst Conditioning” in Chapter 6
		Inspect for excessive nut tension or crimped tubing

System Performance Symptoms

Symptom	Probable Cause	Corrective Action
No response	No gas flow	Check gas source
	Leak in system	Perform leak check
Nonreproducible response for both TIC and TC	Sample volume not constant	Check correct loop size selection under the CONFIGURATION SCREEN
		Check the sample loop for complete filling when sample pump is on
		Inspect pump tubing for leaks or wear; see “Sample Pump” in Chapter 6
	Leak in system	Perform leak check
	Insufficient acid to completely liberate carbon dioxide	Increase acid volume; check acid volume by performing “Calibrating the Acid Pump” procedure in Chapter 6
	Insufficient detect time for high TIC samples	Extend detect time
	Insufficient detect time for complete TC oxidation	Extend detect time
	Injector cap obstructed	Inspect the injector cap
Nonlinear response for TIC and TC	NDIR baseline set too high	Adjust NDIR zero; see “NDIR Zero in Chapter 6
	Carbon mass exceeds linear range of detector	Refer to tables in Chapter 5, “Operation,” for selection of sample volume; reduce sample size or dilute sample
Negative TOC values displayed or printed	Injector cap obstructed	Inspect injector cap operation

Symptom	Probable Cause	Corrective Action
Low response for both TIC and TC	Inaccurate low calibration	See Chapter 5, “Operation,” for calibration procedure
	Inaccurate high sample volume entered	Enter proper sample volume; see Chapter 5, “Operation”
	Wrong sample loop installed	Install proper loop
Low response for TIC with normal or reproducible response for TC	Insufficient acid addition to completely liberate carbon dioxide	Increase acid volume
	Insufficient detect time	Extend detect time
	Improper acid reagent	Confirm that the correct acid solution is in the acid reagent bottle; see Chapter 1 for reagent and materials required
	Faulty acid pump	Check acid pump calibration; see Chapter 6, “Maintenance”
Low response for TC with normal response for TIC	Bad catalyst	Change and/or recondition catalyst
	Injector cap obstructed	Inspect the injector cap
	Combustion furnace not heating	Check temperature on DIAGNOSTICS SCREEN [F7]
	Leak in combustion train	Perform leak check

Symptom	Probable Cause	Corrective Action
High response for TIC and TC	Inaccurate high response factor	Recalibrate TOC Analyzer
	Inaccurate low loop size entered	Enter proper loop size
	System contamination	Perform visual inspection of all surfaces that contact the sample and clean as needed with hot water
		Perform clean water cycling routine described in Chapter 5, “Operation”
Model 1020A will not power up	Model 1020A not plugged into appropriate line voltage	Check power cord connection
		Check power breaker to plug outlet; reset if tripped
	Blown fuse	Contact OI Analytical Technical Support Department

Warning Screens

Warning Screen	Problem/Solution
Warning: Heating Circuit Overtemp	The combustion furnace heater has overheated beyond its set point. Contact the OI Analytical Technical Support Department.
Warning: Low Gas Pressure	The gas supply has dropped below the required pressure to operate the TOC Analyzer.
Warning: Printer Error	The printer is not able to print data. 1020A Firmware will buffer a fixed amount of data after which Firmware will stop trying to print.
Warning: IR Failure: NO OUTPUT	The NDIR output has reached zero (0) and remains at that point.
Warning: Reported Mass Over 275 µgC	The TOC Analyzer has detected a mass of carbon out of its linear range. 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	The TOC Analyzer has no sequence loaded. See Chapter 5, “Operation,” for programming a run.
Warning: Furnace Not At Set Point	The furnace temperature set in the CONFIGURATION SCREEN has not been reached. Allow the furnace to reach the set temperature.
Warning: Method Times/Temps Are Zero	A method parameter is invalid. Press [F5] to check the CONFIGURATION SCREEN.
Warning: IR Baseline Too High	The IR baseline must be below 10,000 to start a run.
Warning: IR Data Error	Hardware problem: contact the OI Analytical Technical Support Department.
Warning: Key Disabled, Use WinTOC	Displayed if keyboard/monitor [F1] or [F2] is pressed while WinTOC is attached and running.
Warning: TC Blank Value Too High	TC Blank area counts >1,000 may indicate decreased catalyst performance. See Chapter 6, “Maintenance” for catalyst cleaning and replacement procedures.

Notes

Chapter 8
Replacement Parts

This chapter lists the order numbers for replacement parts and support items for the Model 1020A 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

PART NAME	PART#	UM	XPND
Boards			
Interface	288035	ea	
Infrared Detector Assembly	295071	ea	
PC104 Controller Assembly	308874	ea	
RAM Memory	289538	ea	
Combustion Heater/Furnace Tube Assembly			
Combustion Heater Furnace - 110 V	311051	ea	*
Combustion Tube Assembly - 680°C	311052	ea	
Combustion Tube Assembly - 900°C	311053		
Sensor - Temp. Manual Reset, 260°F	316182	ea	
Thermocouple - Combustion Furnace	278606	ea	
Tube - Combustion	279067	ea	*
Electronics			
Drive - Floppy Drive (3.5")	266080	ea	
Keyboard - 83-Key	273557	ea	
Monitor - 9" Monochrome	273540	ea	
Power Cord - North America Type	116038	ea	
Power Supply - 25 W, 15 V (PCA)	266742	ea	
Sensor - Temperature	316182	ea	
Ferrules			
⅛" TFZL Flangeless	317545	ea	
18-mm Teflon Tube	224352	10/pk	*
Fittings - Adapters			
⅛ MNPT-F 10-32, Brass, Male/Female	166208	ea	*
10-32 x 1⁄16 Hose, Brass	166191	ea	

Part Name	Part #	U/M	XPND
Fittings			
Coupling ¼"-28 Polypropylene	166274	ea	*
Nut - 18-mm Female, Stainless Steel	224675	ea	
Plug - Brass/Nickel 10-32 O-ring	177502	ea	
Restrictor Element - 0.0025" I.D. 10-32 Blue	280172	ea	
Restrictor Element - 0.0040" I.D. 10-32 White	285296	ea	
Restrictor Element - 0.0018" I.D. 10-32 Black	319330	ea	
Restrictor Element - 0.0030" I.D. 10-32 Orange	319331	ea	
Tee - ⅛" Polypropylene	279125	ea	
Tee - PEEK ¼"-28	319332	ea	
Fittings - Tube End Nuts			
Nut - PEEK ⅛"	319343	ea	
Nut - PEEK ⅛" Red	319344	ea	
Nut - PEEK ⅛" Blue	319345	ea	
Nut - PEEK ⅛" Yellow	319346	ea	
Nut - PEEK ⅛" Green	319347	ea	
Nut - PP ¼"-28, ⅛" Flangeless Green	317479	ea	
Nut - PP ¼"-28, ⅛" Flangeless Yellow	317487	ea	
Nut - PP ¼"-28, ⅛" Flangeless Red	317495	ea	
Nut - PP ¼"-28, ⅛" Flangeless Black	317503	ea	
Nut - PP ¼"-28, ⅛" Flangeless Blue	317511	ea	
Nut - PP ¼"-28, ⅛" Flangeless Clear	317529	ea	
Nut - PP ¼"-28, ⅛" Flangeless Orange	317537	ea	
Nut - PP ¼"-28, 1/16" Tube	307435		
Sample Loops			
25 µL	285650	ea	
50 µL	285668	ea	*
100 µL	285676	ea	*
200 µL	285684	ea	*
400 µL	319334	ea	*

Supplies and Options

Part Name	Part #	U/M	XPND
Analyzer Kits			
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 (.065 mm)	258574	100/pk	*
Vials	210070	250/box	*

Part Name	Part #	U/M	XPND
Autosampler Supplies - 40-mL Vials			
Caps - Open-Hole Screw	296079	72/pk	*
Septa - Teflon-Faced (.065 mm)	258566	100/pk	*
Septa - Teflon-Faced (.065 mm)	173211	50/pk	*
Vials - VOA Autosampler Vials	173196	100/box	*
Chemicals, Reagents, Catalysts			
Ascarite - For Gas Filter	110122	500 g	
Catalyst - Fill Kit	303032	12 g	
Phosphoric Acid, 85%	110080	500 mL	
Phosphoric Acid Solution - 5%, Cleaned	169244	1 L	
Potassium Biphthalate (KHP) - 1000 ppmC	169252	10 mL	
Potassium Biphthalate (KHP) Crystals	136954	500 g	
Quartz Pieces - Crushed Coarse	303024	12 g	*
Sodium Carbonate - 1000 ppmC	169294	10 mL	
Sodium Carbonate, Anhydrous	136962	500 g	
Zirconia Pellets	319422	45g	
Infrared Detector Assembly			
Fitting - Adapter, 10-32 x 1/16" Hose, Brass	166191	ea	
Flow Cell	299081	ea	
NDIR Detector Assembly	179473	ea	
O-ring - Sealing	116400	ea	*
Window - NDIR Cell 20 mm	173287	ea	*
Miscellaneous Parts			
Block Acid Dispense (Model 1051)	277244	ea	
Cap - Humidifier Vial	288985	ea	
Cap - IC Chamber	301630	ea	
Cap - Injector, 0.020	311266	ea	
Cap - Injector, 0.030	311274	ea	
Check Valve/Filter Kit - Reagent Pump	182253	ea	
Clip - Humidifier	263798	ea	
Fan - 12 V 48 CFM	296954	ea	
Fill Tube	303453	ea	
Funnel	286989	ea	*
Gauze - Platinum 80 Mesh 1"	281121	ea	
Manual - Model 1020A Operator's	319406	ea	
Reagent/Water Bottle Cap	281493	ea	
Scrubber Tube, Ascarite	169145	ea	*
Vial - Humidifier	173336	ea	*
Wire - 0.15, 6"	166707	ea	
O-rings			
Buna-N - Infrared Detector	116400	ea	*
Silicone - #15 S70	280198	ea	*

Part Name	Part #	U/M	XPND
Plumbing			
Acid/Water Bottle Assembly	262030	ea	*
Reagent Metering Pump	182550	ea	
Gauge - Pressure 0–60 psi	202325	ea	
Pump Tubing - Gorman-Rupp	277319	ea	
Regulator - 0–60 psi	228023	ea	
Sample Pump - Gorman-Rupp	311290	ea	
POC Module Supplies			
Adapter - 1/16" Female Luer	196386	ea	
Adapter - 1/16" Male Luer	194415	ea	
Fitting - Tee	279125	ea	
POC Module Start-up Kit	317842	ea	
POC Trap	168197	ea	
Printer Supplies/Options			
Interface Cable - Parallel/Centronics Type	273227	ea	
Paper - 9½" x 11"	138546	400 sheets	*
Paper - 9½" x 11"	138554	2500 sheets	*
Ribbon Cartridge - Dot Matrix	178871	ea	*
Tubing and Tube Assemblies			
Tube Assembly - Ascarite Scrubber	169145	ea	*
Tube Assembly - Permeation	264119	ea	*
Tube Assembly Sipper	287938	ea	*
Tubing - Teflon, 1/8" x 0.063 I.D.	147901	ft	*
Tubing - TFE, 1/16" x 0.031 I.D.	145591	ft	*
Tubing - TFE, 1/8" x 0.030 I.D. Clear	112300	ft	*
Tubing - TFE, 1/8" x 0.030 I.D. Green	319326	ft	*
Tubing - TFE, 1/8" x 0.030 I.D. Blue	319327	ft	*
Tubing - TFE, 1/8" x 0.062 I.D. Red	319328	ft	*
Tubing - TFE, 1/8" x 0.062 I.D. Yellow	319329	ft	*
Tubing - TFE, 1/8" x 0.062 I.D. Black	319293	ft	*
Tubing - Viton	313908	in	*
Valves and Valve Assemblies			
Assembly - 3-way Valve	263129	ea	
Valve/Actuator	281147	ea	
Valve - 2-Way Solenoid	319620	ea	
Valve - PP Check	182238	ea	
Pressure Switch	263616	ea	
Drain Valve	296962	ea	
Gas Manifold Assembly	311258	ea	
Chase Acid Pump	182550	ea	
Stud - IC Chamber Cap	295881	ea	

Appendix A
Selecting a Sample Loop

Selecting the correct sample loop is very important; using the wrong sample loop size can cause inaccurate results. Five sample loop sizes are available for the Model 1020A: 25, 50, 100, 200, and 400 µL. Use Table 1 and the following information to select the appropriate sample loop for your analysis.

Table A.1. Sample Loop Volume Applications

Sample Carbon Concentration	Loop Volume	Carbon Mass
0.025–625 ppm	400 µL	0.01–250 µgC
0.5–1,250 ppm	200 µL	0.1–250 µgC
5–2,500 ppm	100 µL	0.1–250 µgC
100–5,000 ppm	50 µL	5–250 µgC
5,000–10,000 ppm	25 µL	125–250 µgC

There is a large overlap of range on various loops (see Figure 1). In order to optimize carbon recoveries, it is recommended that the largest loop size for the expected concentrations be used. This will allow the largest amount of carbon mass available to be measured, thereby increasing the precision and accuracy.

Do not exceed the 250 µgC linear range of the detector. Exceeding this range will prompt a “nonlinear” or “overrange” warning, and the results will be unreliable.

400-µL Sample Loop

Use the 400-µL sample loop to obtain optimum performance at low-level (< 1 ppm) operation with an upper range of 625 ppm. This loop is required for using the POC Module. It is not recommended to use the sample chase for 400-µL sample loop operation.

200-µL Sample Loop

Use the 200-µL sample loop for low range to midrange analysis. This is a good sample loop size for 0.5–1,250 ppm samples. To enhance sample transfer efficiency, it is recommended to use the sample chase for 200-µL sample loop operation.

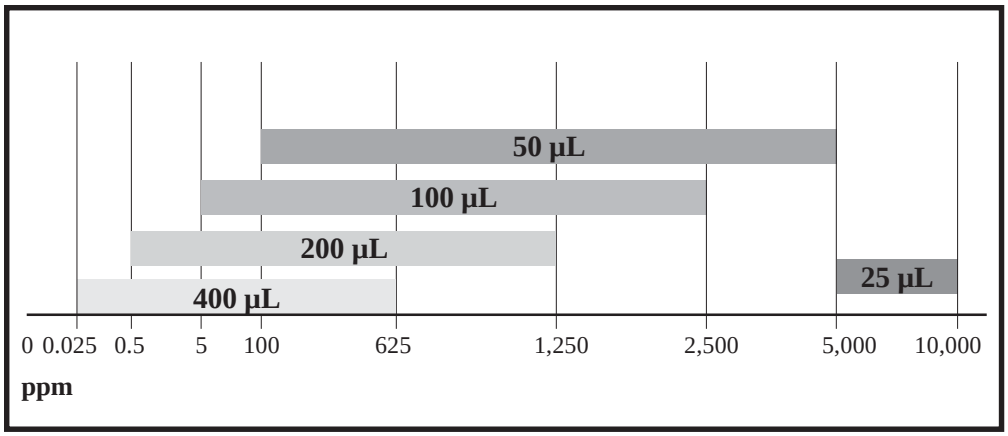


Figure A.1. Sample Range (ppm) Quick-Reference Chart

100-µL Sample Loop

This sample loop comes installed on the Model 1020A from the factory. It is for midrange analysis; optimum range is 5–2,500 ppm. To enhance sample transfer efficiency, it is recommended to use the sample chase for 100-µL sample loop operation.

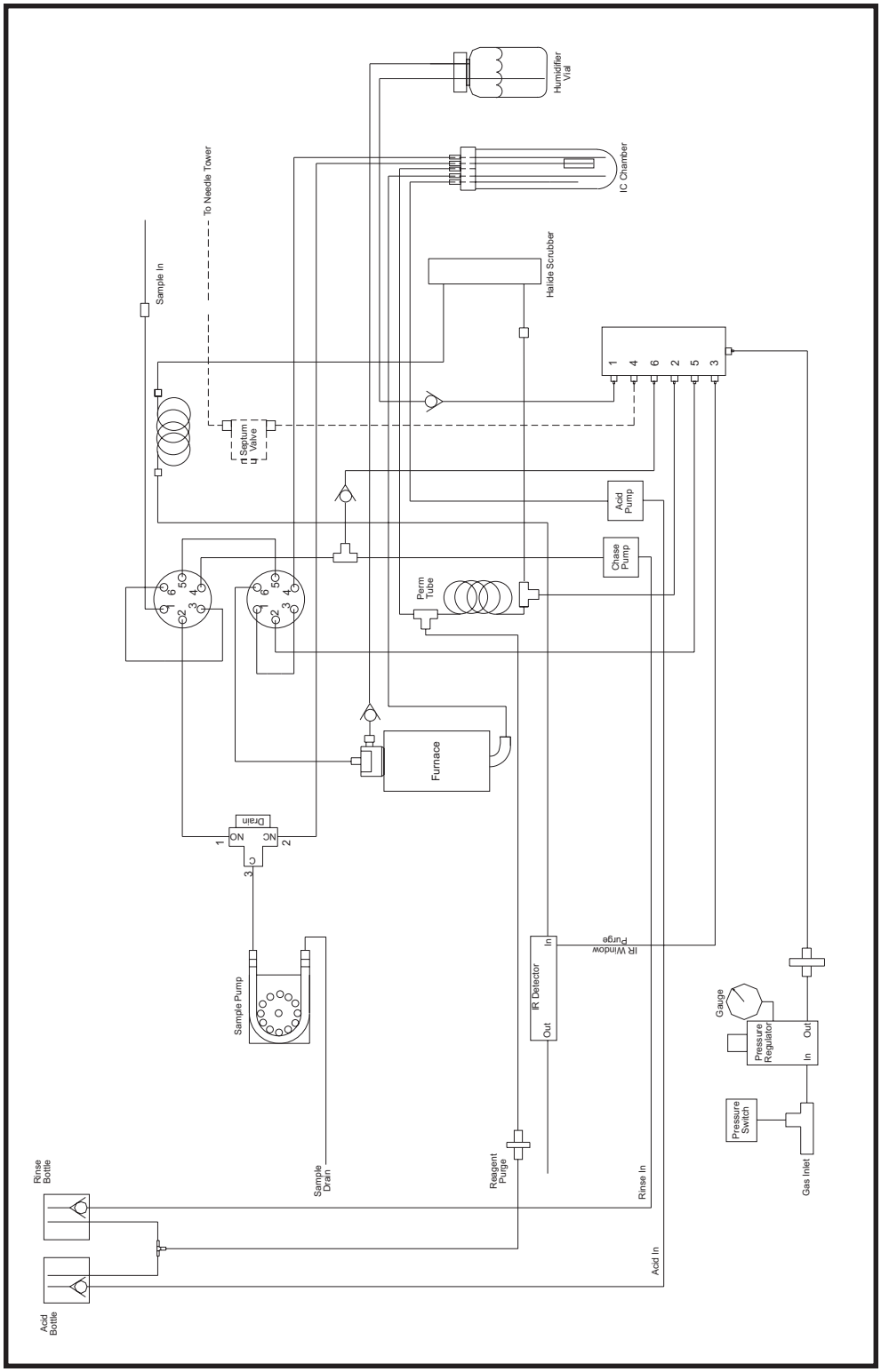
50-µL Sample Loop

Use the 50-µL sample loop for analysis with a range of 100–5,000 ppm. It is highly recommended to use the sample chase for 50-µL sample loop operation to enhance sample transfer efficiency.

25-µL Sample Loop

This loop is for high range (5,000–10,000 ppm) analysis only. Using small bore (0.020 I.D.) tubing to obtain this small volume may require sample preparation to prevent the loop from being obstructed.

Appendix B
Model 1020A Flow Diagram



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