Phoenix 8000

User Manual

DOHRMANN™

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Understanding Safety Warnings

The table below describes symbols used in this manual.

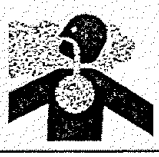
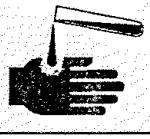
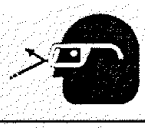
This symbol and/or notice	indicates:
 DANGER	imminently hazardous situation, which, if not avoided, will result in death or serious injury
 WARNING	potentially hazardous situation, which, if not avoided, can result in death or serious injury
 CAUTION	hazardous situation, which, if not avoided, will result in product or property damage and possible injury
	explosion
	hazardous voltage
	fire
	harmful fumes
	corrosive liquid or substance
	high temperature
	risk of eye injury; wear safety glasses
	hazardous or toxic substance
	moving parts can pinch and grab; keep hands and clothing away while power is on

Table 1 Explanation of Symbols

Working Safely

DANGER

To prevent explosion and fire:

- NEVER use hydrogen or other flammable gas with Phoenix 8000.
- Follow the manufacturers' directions for safe handling of gas and chemicals. Also refer to Material Safety Data Sheets for information on specific chemicals.

WARNING

Samples and sample waste may contain hazardous and toxic substances. Follow proper safety and health practices. Also know regulatory limitations before using or disposing of chemicals.

WARNING

To avoid electrical shock:

- Turn off and unplug Phoenix 8000 and the Autosampler before servicing.
- Do not process samples without the panels installed.
- Plug the power cord into a properly grounded outlet.

WARNING

An extension cord may overheat and cause a fire.

Do not plug Phoenix 8000 into an extension cord.

continued

⚠ WARNING  

Acid and persulfate will burn eyes and skin. To prevent injury, wear safety glasses and skin protection when using these chemicals. Refer to Material Safety Data Sheets for detailed information.

⚠ WARNING 

Some parts inside Phoenix 8000 get hot. To prevent burn injury, allow Phoenix 8000 to cool before you remove the panels.

⚠ WARNING

Do not use in a confined space as nitrogen can cause suffocation if accumulated to high levels.

⚠ WARNING

To avoid injury to yourself or damage to Phoenix 8000:

- Do not exceed recommended pressure settings.
- Observe safety regulations when handling pressurized gas. For more information, see Matheson Gases Data Book (available from the Matheson Company, East Rutherford, New Jersey).

⚠ WARNING 

For continued fire protection, replace with same type and rating of fuse.

⚠ CAUTION

Drain system is gravity fed for tray and needle drains. When routing Tygon tubing drain lines, make sure they are sloping downward only. Do not extend tubing into waste bottle by more than 3 to 5 inches. Failure to do so may result in improper drainage of the Phoenix 8000.

continued

⚠ CAUTION

To prevent damage to the infrared detector:

- Place Phoenix 8000 in an area that is free from direct sunlight and abrupt temperature changes.
- Avoid exposing Phoenix 8000 to corrosive or combustible gases.

⚠ WARNING

Extremely high voltage is present throughout the Phoenix 8000 unit. Turn off and unplug Phoenix 8000 before servicing.

Note: Excessive electromagnetic interference (EMI) may cause the infrared detector to operate improperly. Do not place Phoenix 8000 near high frequency furnaces or other sources of EMI. If you must place Phoenix 8000 near an EMI source, use a separate power line. If you have problems, please call Tekmar-Dohrmann Service. This unit has passed EMC Directive requirements; EN50081-1 and EN50082-1.

Introduction to TOC Analysis

Total organic carbon (TOC) analysis grew from the need to analyze wastewater and municipal water for organic matter. Groundwater is one of our most important sources of drinking water. The need to protect it and establish criteria for screening and measuring contaminant levels was recognized when Congress enacted the Resource Conservation and Recovery Act (RCRA) in 1976. This act requires operators of waste disposal sites to monitor their groundwater on a quarterly basis for organic carbon levels and organic halide levels.

TOC analyzers are also widely used in monitoring the quality of process water in the semiconductor and pharmaceutical industries. Because organic material can cause contamination, TOC analysis is also performed to protect process equipment such as boilers, turbines, and purification devices. Furthermore, TOC levels in solids such as soils, clays, and sediments are of increasing interest.

TOC analyzers can measure total carbon (TC), total organic carbon (TOC), inorganic carbon (IC), purgeable organic carbon (POC), and nonpurgeable organic carbon (NPOC). TOC measurement involves 1) oxidizing organic carbon in a sample, 2) detecting and quantifying the oxidized product (CO_2), and 3) presenting the result in units of mass of carbon per volume of sample.

Figure P-1 and the text that follow give an overview of commonly measured parameters.

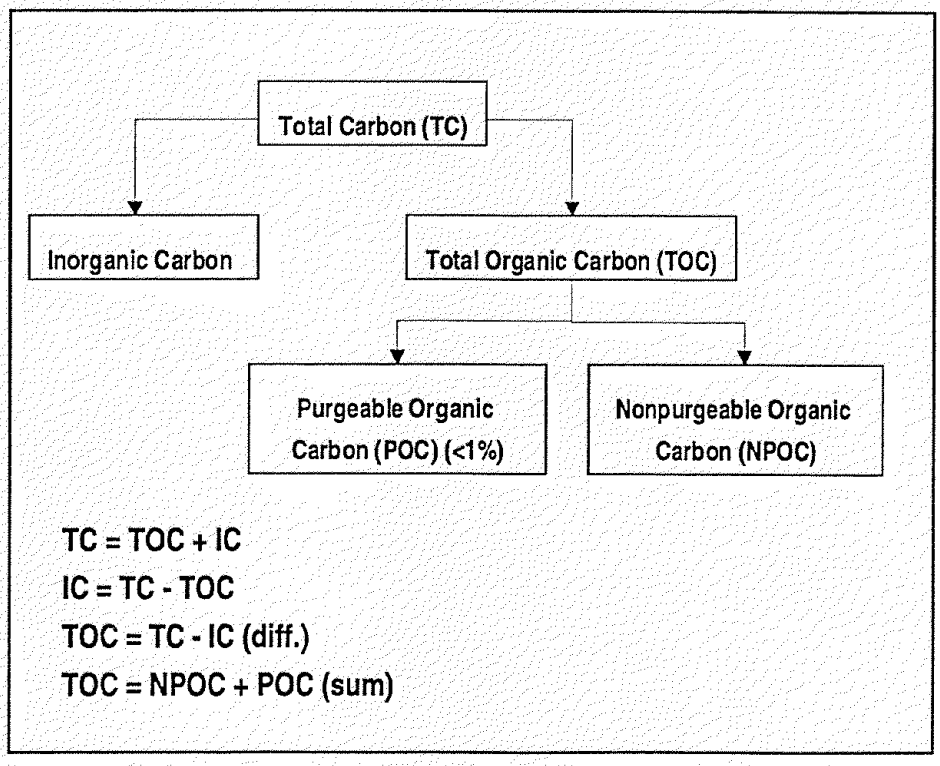


Figure P-1 Carbon Analysis Diagram

Total Carbon (TC)

TC is a measure of all the carbon in the sample, both inorganic and organic, as a single parameter. Generally, the measurement is made by placing the sample directly into the analyzer without pretreatment.

Total Organic Carbon (TOC)

TOC is the sum of all the organic carbon in the sample. There are three ways to measure TOC: directly, by difference, and by sum.

TOC Measurement Directly

In the direct approach, inorganic carbon is first removed by acidification and sparging, and the remaining carbon is measured as TOC. Inorganic carbon and purgeable organic compounds are not recovered for further analysis in this approach. However, since POC generally represents one percent or less of total carbon in a sample, it is considered negligible.

TOC Measurement by Difference

This approach requires two analyses: one to measure TC, and one to measure IC. The difference between these two measurements is rigorously TOC.

TOC Measurement by Sum

This approach measures nonpurgeable organic carbon (NPOC) and purgeable organic carbon (POC). The sum of these measurements is rigorously TOC.

Inorganic Carbon (IC)

Inorganic materials are those comprised entirely of carbon and oxygen. IC includes carbonate, bicarbonate, and dissolved carbon dioxide. IC is analyzed in liquid samples by acidifying with an inorganic acid to pH 3 or lower, and then sparging with a stream of inert gas. The acidification converts carbonates and bicarbonates to carbon dioxide, which is then removed along with dissolved CO_2 by the gas stream and measured to provide an IC value.

Purgeable Organic Carbon (POC)

POCs are volatile and semivolatile organic materials sparged from a sample. However, these materials are generally less than one percent of total carbon in a sample.

Analyzing TOC

As mentioned earlier, TOC measurement involves 1) oxidizing organic carbon in a sample, 2) detecting and quantifying the oxidized product (CO_2), and 3) presenting the result in units of mass of carbon per volume of sample. Each step in this process is discussed below.

Oxidation

The Phoenix 8000 uses wet chemical methods for oxidation. Specifically, the process utilizes persulfate oxidation along with UV irradiation. The sample is simultaneously exposed to persulfate ions and to UV radiation, which produces a highly reactive sulfate and hydroxyl-free radicals.

The CO_2 produced is swept by a stream of gas such as nitrogen to the detector.

Detection and How Results are Displayed

During an analysis, CO_2 levels are measured by a nondispersive infrared (NDIR) detector. The detector is sensitive to the absorption frequency of carbon dioxide and provides a signal proportional to the instantaneous concentration of CO_2 in the carrier gas flowing through it. The detector output signal is linearized and provides a reading of TOC. As the carbon dioxide is transferred from the UV reactor, it is continuously monitored by the NDIR detector. This response is displayed on the video screen as a real time strip chart recording of the output. The linearized signal is integrated and referred to stored calibration data and the carbon concentration in the sample is calculated to display carbon concentration in parts-per-million (ppm).

1.1 Description of the Phoenix 8000

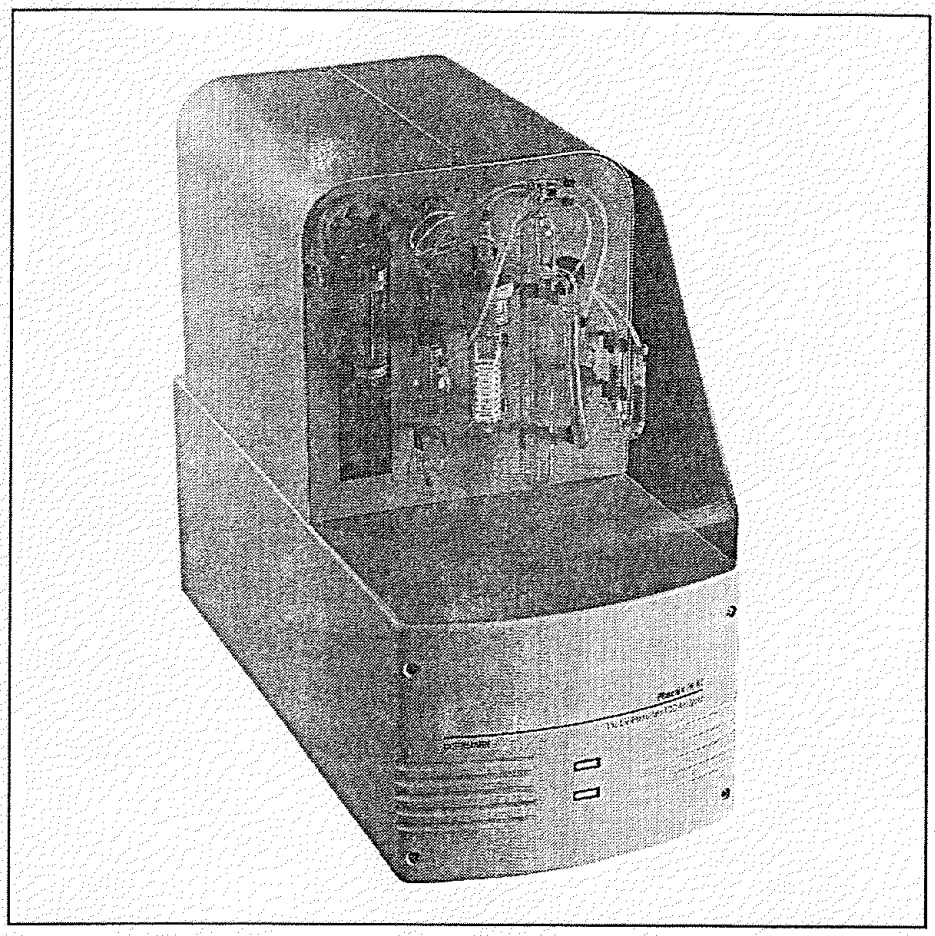


Figure 1-1 Phoenix 8000

Dohrmann's Phoenix 8000 is a reliable, easy-to-use, easy-to-learn TOC analyzer that offers superior performance and delivers accurate results. Parts-per-million (ppm) or parts-per-billion (ppb) carbon concentrations are calculated effortlessly through TOC Talk, the Phoenix 8000 software.

Phoenix 8000 is comprised of the following:

- √ Syringe - for sample and reagent introduction
- √ Sparger - for inorganic analysis and sample preparation for TOC analysis
- √ UV Cell - for carbon oxidation
- √ Gas/Liquid Separator - for moisture removal
- √ Mist Trap - for moisture removal
- √ Permeation Tube - for moisture removal
- √ Chlorine Scrubber - for halogen removal
- √ NDIR Detector - for carbon dioxide detection

The following are accessories for Phoenix 8000:

- Printer
- STS 8000 Autosampler
- IC Sparger Flowmeter
- UV Reactor Flowmeter

See Chapter 2, "Parts Description," for a general overview of each Phoenix 8000 component.

User interface is achieved through TOC Talk, the Phoenix 8000 software, which offers effortless monitoring and calculations in a Windows 95 platform (fully compatible with Windows 3.1). Guided by menu screens, users can calibrate, set up, run, print results, and repeat an analysis with great accuracy, precision, and throughput. Much of the preliminary work in setting up a run is automated. The user clicks on the method name and the associated method details appear—just clicking on **Start** begins the run.

The following are some of the features of Phoenix 8000 software, TOC Talk:

- Multiple Method Storage
- Analog Display of Peaks
- Continuous Scrolling of Results
- Multi-Point Calibration
- Plotted Calibration Curve and Statistics
- Diagnostics

The Phoenix 8000 complies with EPA Methods 415.1 and 9060A; Standard Method 5310C; ASTM D4779 and D4839; and USP TOC Method.

1.2 Unpacking the Phoenix 8000

This section explains how to unpack Phoenix 8000. Failure to follow instructions could void your warranty for parts damaged in shipment.

1. Refer to "Phoenix 8000 Unboxing the Unit" instructions. They are shipped with the unit, in the box.

2. Place Phoenix 8000 on a dry workbench strong enough to support its weight of 95 pounds. If you are using the autosampler, the table must be able to support an additional 40 pounds.

3. Sufficient space should be allocated around the instrument to enable convenient user access. Keep in mind that space must be allocated to the right or rear of the unit for water, reagent, and waste containers. Also keep in mind that you will need to connect Phoenix 8000 to a nitrogen gas supply. Additional space to the left of the unit will be required for optional accessories (i.e. autosampler). It is recommended that you allot 2 feet each for the Phoenix 8000 unit, PC and optional autosampler.

4. The work environment should be free of corrosive or explosive vapors and should have a reasonably constant temperature. Air conditioning is not required.

5. Carefully examine Phoenix 8000. If it is damaged, notify the shipping carrier and Tekmar-Dohrmann immediately. Do not continue installation. Return Phoenix 8000 only after a Tekmar-Dohrmann service representatve has issued an authorization number.

6. Save all shipping materials.

> ⚠ **CAUTION**
>
> **To avoid damage to Phoenix 8000, do not place any liquids on top of unit.**

1.3 Technical Specifications

Phoenix 8000

Chemistry:	Oxidation by UV-Persulfate
Detector:	Nonlinear, Nondispersive Infrared (NDIR)
Analysis Type (Modes):	TC TOC (NPOC) TC-IC IC
Range:	Limit of Detection: 2 ppb Maximum Measurable Concentration: 10,000 ppm (sample volume and dilution dependent)
Precision:	± 2%, ± 1 ppb or ± 0.02 µgC RSD over 7 samples, whichever is greater
Sample Size:	500 µl to 20 ml
Analysis Time:	4 to 8 minutes, typical
Liquid Handling:	Syringe pump, 8-port distribution valve Dilution method provided for range 200ppm to 10,000ppm Auto-rinsing from sample and/or rinse water
Sample Introduction:	Automatic syringe injection STS 8000 Autosampler
Controller:	PC, Interface through Windows® (3.1 or 95) Linearization of detector signal
Data Handling:	Spreadsheet reports transferable to Microsoft® Excel® Real-time display of curves Ability to store customized individual test methods Priority samples via scheduled interrupt
Calibration:	Multi-point and auto-blanking

Official Methods and Principal Applications:	EPA 415.1 and 9060A Standard Method 5310C ASTM D4779 and D4839 Cleaning Validation USP TOC Method 643 Boiler Feed Water Cooling Water Drinking Water Surface Water Ground Water
Certification:	CE EMC EN 50081-1 and EN 50082-1
Utility Requirements:	Voltage: 100/120/230 VAC (±10%) Frequency: 50/60 Hz (excluding NDIR) Power: 368 VA
Dimensions:	cm 30.5 W x 63.5 D x 56 H in 12" W x 25" D x 22" H 54.4 kg (120 lb.) shipping weight
Gas Supply:	99.98% pure nitrogen 99.999% pure nitrogen for trace levels 99.98% Zero grade air Balston / Whatman TOC Gas Generator
Gas Pressure:	30 to 35 psi (206.7 to 241.2 kPa)

STS-8000 Autosampler

Sampler Changer Type:	XYZ robot with stationary rack design
Positioning Performance:	Accuracy ±1mm in XYZ dimensions. Repeatability ±0.25 mm in XYZ dimensions.
Septum Piercing:	Available with septum piercing kit. Has vertical punch strength of 3.8 kg (8.38 lbs.)
Rinsing:	Auto-rinsing from sample and/or rinse water via build-in rinse station.
Rack Selection:	(2) 77 position trays for 25ml culture tubes (18 x 150 mm) (2) 42 position trays for 50-60* ml culture tubes (25 X 150 mm) (2) 35 position trays for 40ml VOA vials (28 X 95 mm) (2) 12 position trays for 4 oz. (125 ml) Boston Round bottles (48 X 117 mm) * *screw cap tube has 50 ml capacity to neck. Disposable tube has approximate overflow capacity of 60 ml*
Dimensions:	cm 53.5 W x 43.7 D x 37.1 H in 21.1 W x 17.2 D x 14.6 H 17.7 kg (39 lbs)
Electrical: Voltage:	100/120/230 VAC (±10%) Frequency: 50/60 Hz Power: 200VA
Certification:	UL, CSA, and CE EMC EN50081-1 and EN 50082-1

2.1 Overview

This chapter briefly describes Phoenix 8000's internal and external parts. The figures below show the locations of the parts.

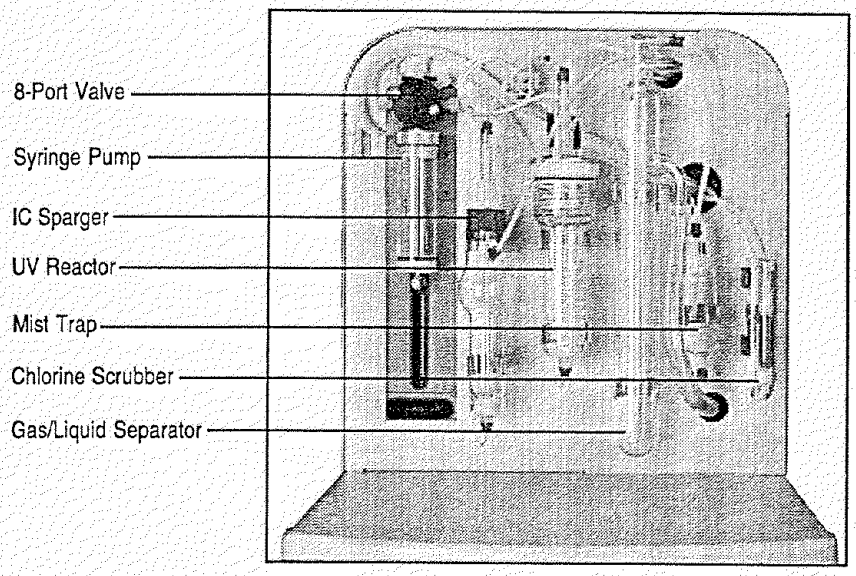


Figure 2-1 Front View of Phoenix 8000

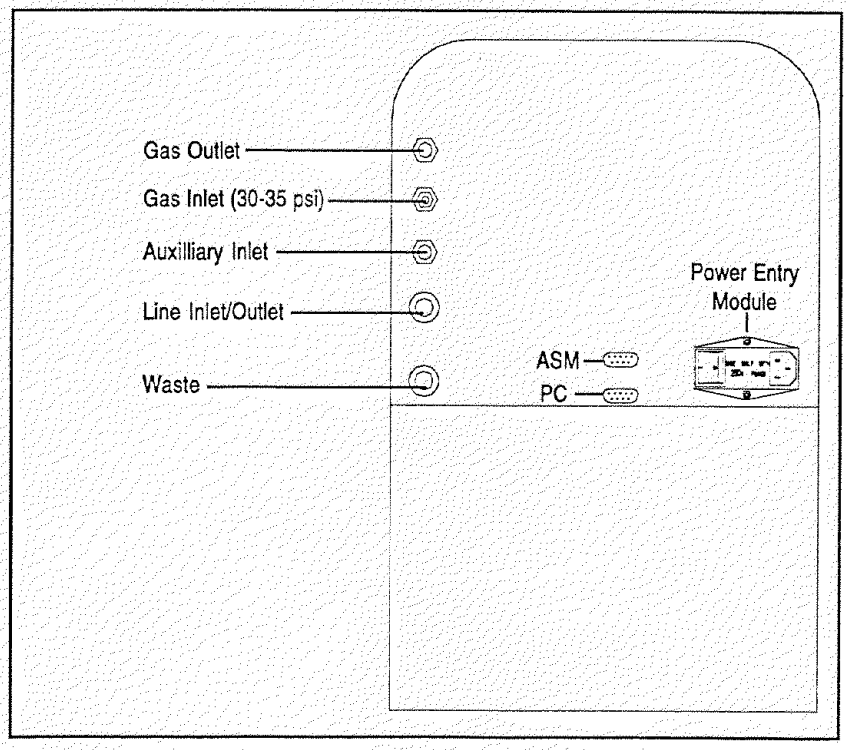


Figure 2-2 Rear View of Phoenix 8000

2.2 Syringe Pump

The syringe pump is a precision measuring instrument that aspirates and dispenses fluid. The syringe pump contains electronics, a syringe, and stepper motors that drive valves. The electronics control the motors and communication between the syringe pump and microcontroller.

The syringe pump can dispense 40 µL to 25 ml (± .1%) of sample or reagent.

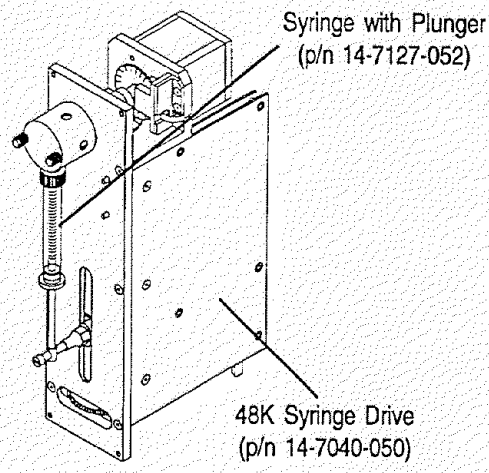


Figure 2-3 Syringe Pump (p/n 14-7071-052)

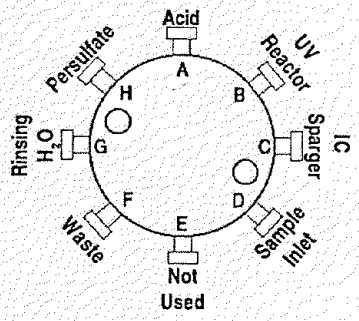


Figure 2-4 8-Port Valve (p/n 14-7040-050)

The syringe pumper turns an 8-port Teflon valve. Each port on the valve is fitted with a valve washer before insertion of relevant tubing fitting to ensure an airtight seal. The port assignments for this valve can be found on Figure 2-4.

2.3 IC Sparger

The sparger is a glass vessel that holds the sample while Phoenix 8000:

- purges the sample of inorganic carbon (IC) and purgeable organic carbon (POC), and
- prepares the sample for TOC analysis.

Gas flows through the sparger, removing the IC from the sample. The Phoenix 8000 can detect IC in IC mode or send it to vent while preparing for TOC mode.

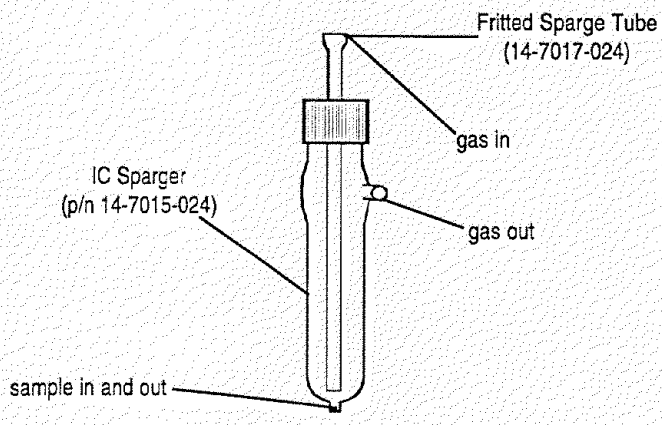


Figure 2-5 Sparger

2.4 UV Reactor

The UV reactor is composed of a glass vessel and an ultraviolet (UV) light source. Phoenix 8000 introduces the sample and persulfate reagent into the UV reactor. The persulfate reagent, combined with UV light, oxidizes carbon in the sample.

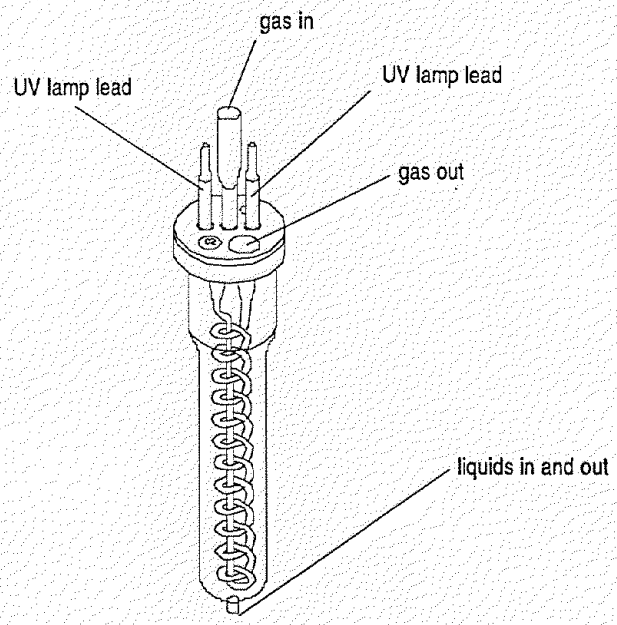


Figure 2-6 UV Reactor (p/n 14-7194-000)

2.5 Chlorine Scrubber

The detector, which measures carbon dioxide, can be damaged by halogen. To prevent analytical errors, the chlorine scrubber removes chlorine from the carbon dioxide before it enters the detector. The chlorine scrubber is a long glass tube filled with Pyrex wool and tin and copper granules.

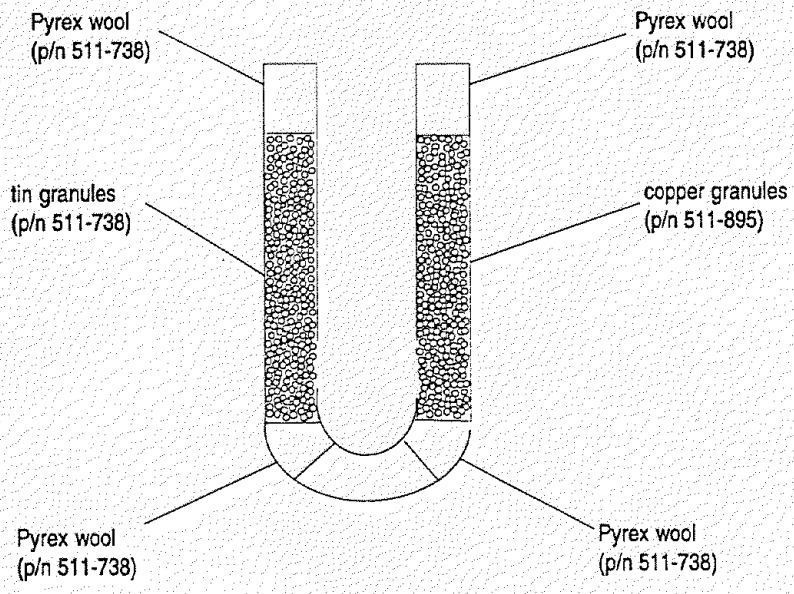


Figure 2-7 Chlorine Scrubber (p/n 14-7014-024)

2.6 Moisture Control System

The detector, which measures carbon dioxide, can confuse water vapor and CO_2, so Phoenix 8000 removes moisture from the sample.

Phoenix 8000's moisture control system consists of a gas/liquid separator, mist trap, and permeation dryer.

The UV reactor (where carbon in the sample is converted to carbon dioxide) generates low heat. Carrier gas sweeps CO_2 and water vapor out of the UV reactor. The sample then travels through tubing where condensation may occur.

The sample enters the gas/liquid separator. This device removes most of this condensation, isolating the carbon dioxide from the moisture.

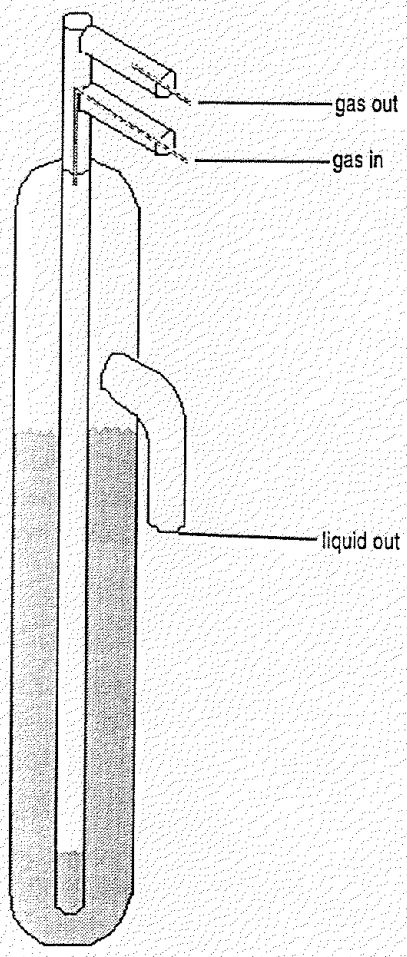


Figure 2-8 Gas/Liquid Separator (p/n 14-7029-024)

Next, the carbon dioxide travels through a mist trap, where additional moisture is removed.

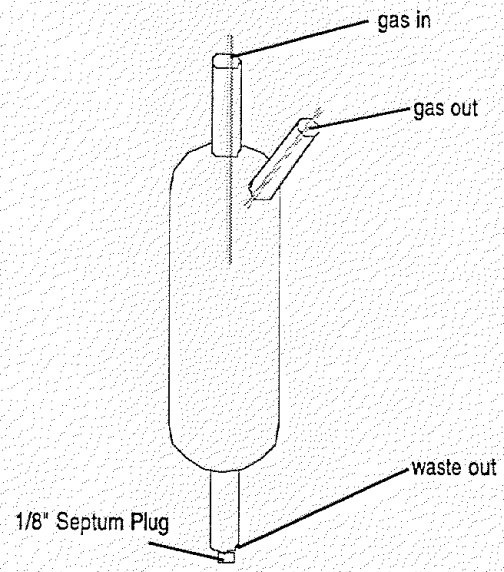


Figure 2-9 Mist Trap (p/n 885-143)

Then the carbon dioxide passes through a gas permeation tube to further remove moisture. The tube is sealed into an impermeable shell, which has openings adjacent to the sample inlet and product outlet. When a wet gas stream flows through the the tube and a countercurrent dry gas stream purges the shell, water vapor molecules are transferred through the walls of the tubing.

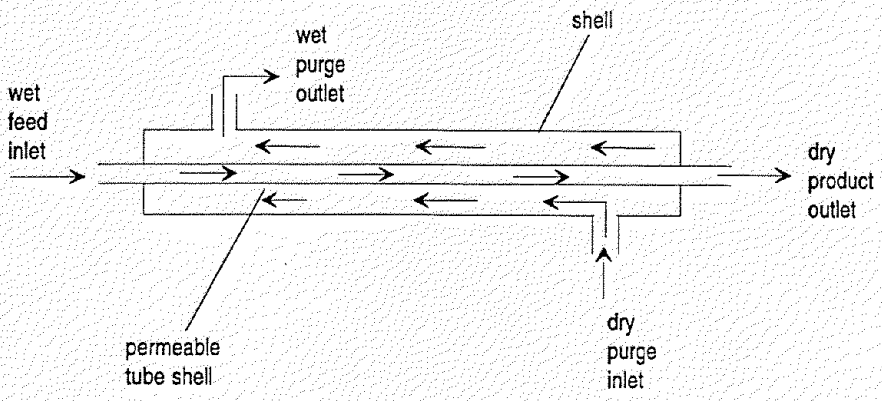


Figure 2-10 Permeation Tube (p/n 090-825)

2.7 Nondispersive Infrared Detector

Phoenix 8000 converts carbon in the sample to carbon dioxide. The nondispersive (single beam) infrared detector uses electromagnetic radiation or infrared energy to measure this carbon dioxide. This measurement is proportional to the carbon in the sample. The following paragraphs briefly explain how the nondispersive infrared (NDIR) detector works:

Inside the detector, a beam of infrared energy first passes through a sample cell; then two detector cells (*Figure 2-10*). The sample cell receives the sample gas or carbon dioxide. The two detector cells (front cell and rear cell) are filled with the carbon dioxide that the detector must measure. When using the NDIR for carbon analysis, both of these detector cells are filled with carbon dioxide. Carbon dioxide shows a unique adsorption spectrum when infrared energy passes through it. Therefore, the NDIR can separate it from other gases.

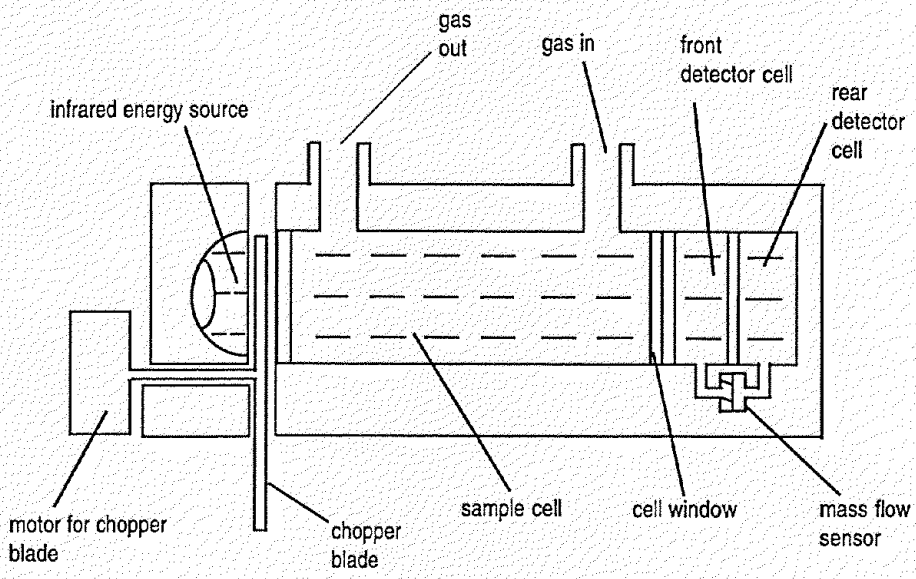


Figure 2-11 Nondispersive Infrared Detector (NDIR) (p/n 010-905)

When the infrared beam enters the sample cell, the sample gas absorbs some of the infrared beam's energy. The infrared beam enters both detector cells, where carbon dioxide gas absorbs more of the beam's energy. This causes the gas in both cells to heat and expand. The gas in the front detector cell absorbs more energy than the gas in the rear detector cell. This is because the energy from the infrared beam is stronger when it enters the front detector cell. Since the amount of energy reaching the rear cell is less than the energy reaching the front cell, the expansion is unequal. This unequal expansion causes gas to flow between the two detector cells.

A rotating chopper blade interrupts the infrared beam at regular intervals. As a result of these interruptions, the beam pulses. Internal pressure, as well as the flow rate of the gas that is traveling between the two detector cells, rises and falls to the pulses' rhythm. A mass flow sensor detects and converts the rhythmic flow into an electrical signal. The NDIR measures the resulting voltage and displays the measurement on its front panel.

2.8 Flow Restrictors

Four flow restrictors maintain the flow rate of the supply gas as follows:

1. 200 ml/min to the UV reactor (p/n 090-099)
2. 200 ml/min to the IC sparger (p/n 090-099)
3. 100 ml/min to the permeation tube (p/n 090-097)
4. 20 ml/min to purge the NDIR (p/n 090-093)

NOTE: When you turn off the gas (valve 1), 20 ml/min of carrier gas continues to purge CO_2 from the NDIR.

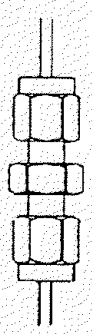


Figure 2-12 Flow Restrictor

2.9 Standard Flowmeters

The Phoenix 8000 is equipped with one standard flowmeter (p/n 14-7195-000). This NDIR flowmeter monitors the flow rate of carrier gas exiting the NDIR.

2.10 Optional Flowmeters

Two flowmeters, which are accessories (p/n 14-7195-000), monitor the flow rate of the gas. One flowmeter monitors the flow rate of the gas entering the sparger. The other flowmeter monitors the flow rate of the gas entering the UV reactor.

2.11 Optional STS 8000 Autosampler

The optional autosampler (p/n 14-7046-000) is an XYZ robot that automates sample handling procedures. It allows the user to increase throughput and operate the analyzer unattended for many hours.

The autosampler tray holds 2 racks; each rack can hold up to 77 25ml and 42 50-60ml culture tubes, 35 40ml VOA vials and 12 4oz. (125ml) Boston Round bottles. All vials and bottles are fitted with pierceable Teflon-backed septum caps.

The autosampler rinses the inside and outside of the needle via a dedicated rinse station.

The arm speed of the *x* and *y* axis is 250 mm/sec.

Making it even more functional, the autosampler also allows different programming for samples on the same rack.

3.1 Overview

This chapter tells you how to:

- make electrical connections
- prepare reagents
- swage a nut and ferrule
- connect Phoenix 8000 to gas, water, and reagent supplies
- connect drain lines.

3.2 Making Electrical Connections

Power requirements for Phoenix 8000 are as follows: 100/120/230 VAC (±10%), 50/60 Hz, 750 VA, 2574 Btu/hour. It is best to use a power line dedicated to instrument use only. Surge protection is highly recommended.

⚠ WARNING

Do not plug Phoenix 8000 into an extension cord. An extension cord may overheat and cause a fire.

Please note that the NDIR detector requires approximately two hours to warm up before operating.

NOTE: Once electrical connections are made, it is highly recommended that you leave the unit powered on and place Phoenix 8000 on standby mode through the software. This option will power down all components of the unit except the NDIR detector—eliminating the need for warm up before each use.

RS-232 straight through 9-pin cable connectors allow you to link Phoenix 8000 to the computer and accessories. Locate the RS-232 cable. Attach the male end of the cable to the appropriate port on the rear panel of Phoenix 8000. Tighten the retaining screws. Attach the other end of the cable to the appropriate port (com 1 or com 2 only) on the rear panel of the computer. Again, tighten the retaining screws.

3.3 Preparing Reagents

To prepare reagents, gather the following supplies:

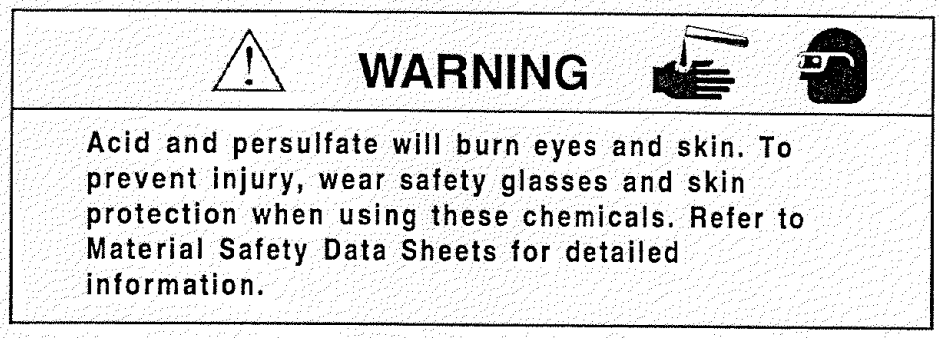
- bottles shipped with unit:

  (2) 1 liter bottles for acid and persulfate (p/n 21-0595-000)

  (1) 2 liter bottle for DI H_2O (p/n 14-7019-000)

- ultra pure water (distilled water treated with activated carbon resin)
- phosphoric acid (H_3PO_4) 85%
- sodium persulfate ($Na_2S_2O_8$) 98+%

To prevent organic contamination, wash bottles thoroughly with hot, soapy water and rinse at least three times with ultra pure water before using.



NOTE: Put on safety glasses and protective clothing before proceeding to the next step.

To Prepare 0.5 Liter 21% Acid Reagent:

- Measure 74 ml 85% phosphoric acid (H_3PO_4) into rinsed bottle.
- Add 375 ml ultra pure water.

To Prepare 0.5 Liter 10% Persulfate and 5% Phosphoric Acid Reagent Mixture:

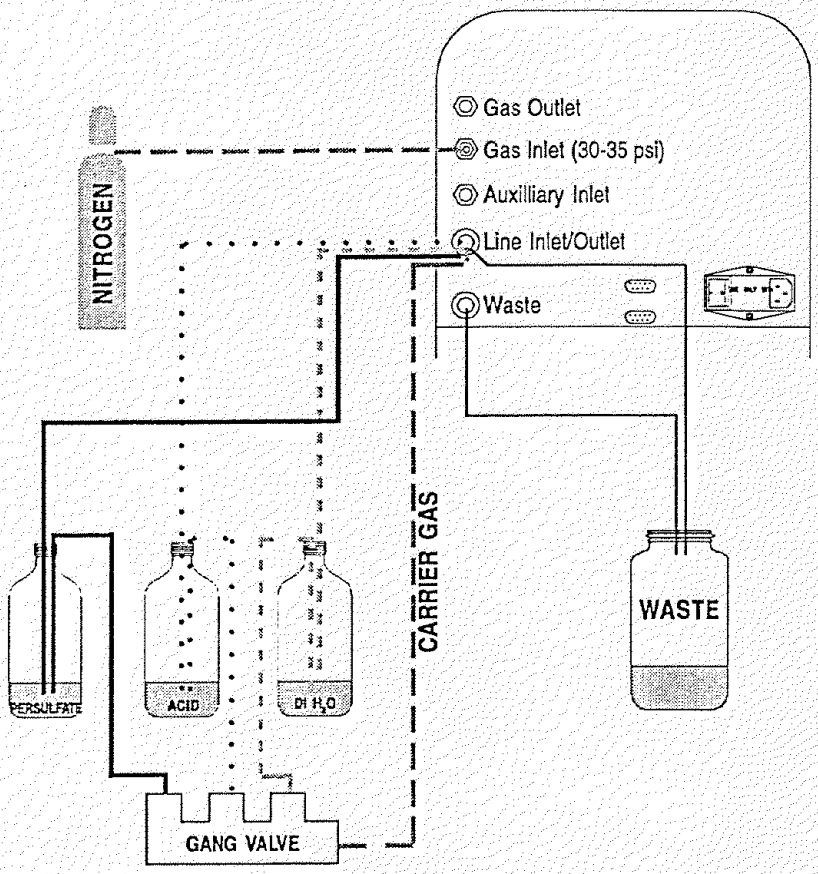
- Measure 50 g 98+% sodium persulfate ($Na_2S_2O_8$) into rinsed bottle.
- Add 15 ml 85% phosphoric acid (H_3PO_4).
- Add 425 ml ultra pure water.

For best results, store prepared solutions away from direct sunlight and use the persulfate reagent within one week and the phosphoric acid reagent within one month.

3.4 Understanding Gas, Water, and Reagent Connections

Figure 3-1 illustrates the gas, water, and reagent connections that need to be made before operating Phoenix 8000.

Specific instructions for each connection are given in the sections that follow.



YELLOW LINES
RED LINES
CLEAR LINES
BLUE LINES

Six tubes exiting opening:

1. **clear tube to deionized water**
2. **red tube to acid**
3. **blue tube to persulfate**
4. **yellow tube: nitrogen out teed to acid, persulfate, and deionized water**
5. **black tube for dispensing syringe waste**
6. **vent gas (no attachments)**

One tube exiting bottom opening to waste

Figure 3-1 Rear Panel: Gas, Water, and Reagent Connection Diagram

3.5 Tools and Supplies Needed for Connections

Before you begin making connections, gather the following tools and supplies:

- nuts and ferrules supplied with unit
- 1 1/8" open end wrench
- 1/2" open end wrench
- 7/16" open end wrench
- tank of pure nitrogen gas equipped with a two-stage regulator for steady delivery of gas at 30 to 35 psi (206.7 to 241.2 kPa)
- large bottle or beaker to collect waste.

3.6 Swaging a Nut and Ferrule onto Tubing

To connect Phoenix 8000 to gas supplies, you must *swage* nuts and ferrules onto tubing; then connect the tubing to Phoenix 8000. This section instructs you how to complete this task correctly. If the nuts and ferrules are the wrong sizes or are not properly swaged, leaks can occur.

All the teflon tubing requires either a one-piece plastic ferrule or a two-piece metal ferrule.

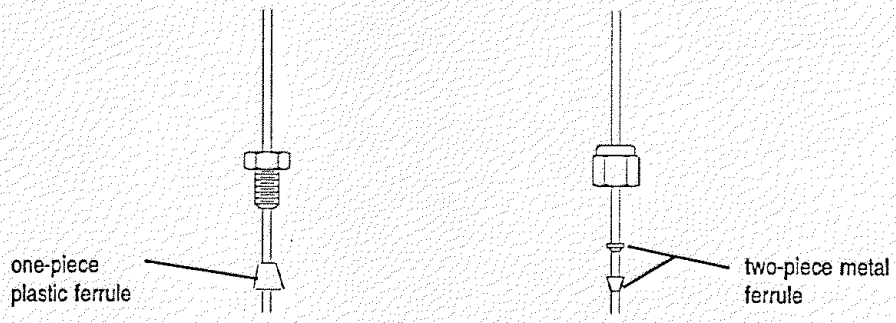


Figure 3-2 Placing Nuts and Ferrules onto Tubing

1. Slide the nut onto the tubing.
2. Slide the ferrule onto the tubing, orienting the smallest end of the ferrule toward the nut. Allow about 3 mm (1/8 in.) of tubing to extend past the end of the ferrule.
3. Insert the tubing into the designated connector on Phoenix 8000.
4. Tighten the nut with your fingers. For metal ferrules (such as to the gas supply), use a wrench to tighten the nut further. Turning the nut 1/4 turn (90°) to 1/2 turn (180°) is usually adequate. However, the amount of force you need to apply can vary, depending on the friction between the nut and threads, as well as the composition and thickness of the tubing or line.

NOTE: To check if a nut and ferrule have been properly swaged, loosen the nut and pull on the ferrule. The ferrule should not easily slide.

⚠ CAUTION

To prevent damaging the metal nuts and ferrules, do not tighten them over 3/4 turn (270°). Once swaged onto tubing, you may need to tighten a nut only slightly to eliminate a leak. If leaking persists, look for other causes of the leak.

3.7 Connecting Phoenix 8000 to a Gas Supply

1. Before connecting Phoenix 8000 to a gas supply, read section 3.6, *Swaging a Nut and Ferrule onto Tubing*.
2. Turn the pressure on the two-stage gas regulator to zero psi. (This prevents a sudden burst of pressure from damaging parts.)
3. Locate the fitting labeled "Gas In" on the back of the unit. Using a nut and ferrule, connect your tubing from the gas supply tank to this fitting. Do not overtighten the nut.
4. **Do not turn on the gas supply at this time.** Go to the next section to continue making connections before operating Phoenix 8000.

⚠ WARNING

Do not use in a confined space as nitrogen can cause suffocation if accumulated to high levels.

⚠ DANGER

To prevent explosion and fire:

- **NEVER use hydrogen or other flammable gas with Phoenix 8000.**
- **Follow the manufacturers' directions for safe hand ling of gas and chemicals. Also refer to Material Safety Data Sheets for information on specific chemicals.**

⚠ WARNING

To avoid injury to yourself or damage to Phoenix 8000:

- **Do not exceed recommended pressure settings.**
- **Observe safety regulations when handling pressurized gas. For more information, see Matheson Gases Data Book (available from the Matheson Company, East Rutherford, New Jersey).**

3.8 Connecting Phoenix 8000 to Water Supply

Organic-free deionized or distilled water (50 ppbC or lower is required for trace analysis) is recommended for proper operation of the Phoenix 8000.

NOTE: For best results, change water on a daily basis.

Locate the bottle labeled DI water and place it to the right of the unit.

Please refer to the connection diagram (Figure 3-1) for the location of the proper tube to place into your water supply. It is the 1/8" clear tube in the group of five exiting the back of the unit.

Insert this tubing to the bottom of the water container.

3.9 Connecting Phoenix 8000 to Acid and Persulfate Supplies

Follow the steps below to connect acid and persulfate containers to Phoenix 8000.

1. If you have not already done so, refer to section 3.3, *Preparing Reagents*, and prepare your reagents.
2. Place containers with prepared reagents to the right of Phoenix 8000.
3. The acid and persulfate containers each have a cap with holes for inlet and outlet connections and vent holes.
4. Referring to Figure 3-1, locate the **red** 1/8" tube (p/n 14-7108-002) exiting the back of the unit. Place this tube into the appropriate hole in the container labeled **acid** insuring that tube is placed near the bottom.
5. Referring to Figure 3-1, locate the **blue** 1/8" tube (p/n 14-7111-002) exiting the back of the unit. Place this tube into the appropriate hole in the container labeled **persulfate** insuring that tube is placed near the bottom.

A stream of gas flowing through acid and persulfate will ensure the solutions' purity. The next two steps explain how to connect a nitrogen supply to the solutions.

6. Referring to Figure 3-1, locate the **yellow** 1/8" tube (p/n 14-7112-002) exiting the back of the unit.
7. Attach line to 3-way gang valve (p/n 14-7025-050).
8. Attach gang valve uotputs to acid persulfate and DI water bottles insuring that tube is placed near the bottom.

Do not turn on the gas supply at this time. Go to the next section.

WARNING

Acid and persulfate will burn eyes and skin. To prevent injury, wear safety glasses and skin protection when using these chemicals. Refer to Material Safety Data Sheets for detailed information.

3.10 Filling Gas/Liquid Separator

Locate the gas/liquid separator and disconnect the three tubes. Remove the separator from the unit. Fill the gas/liquid separator with ultra pure water until the liquid is even with the bottom of the sidearm. Carefully place back onto the unit and reconnect the tubing.

NOTE: It is recommended that you add .25 mL phosphoric acid to the DI water in the gas/liquid separator to prevent contamination.

3.11 Connecting Phoenix 8000 to Drain Line

Follow the steps below to connect the drain line to Phoenix 8000:

1. Place waste container of your choice (not supplied) to the right of the unit.
2. Referring to Figure 3-1, locate the 1/2" clear tube and the 1/8" black line (p/n 14-7109-002) tied together exiting the back of the unit. Place these tubes into the waste container.

NOTE: It is important that tube does not have contact with waste water. The tube must remain in the air above the waste water.

3. Adjust the pressure to these lines as needed to insure adequate carrier gas flow to the acid persulfate and DI water bottles.

4.1 Overview

This chapter tells you how to connect the autosampler to Phoenix 8000. *Figure 4-1* shows a possible configuration of the autosampler.

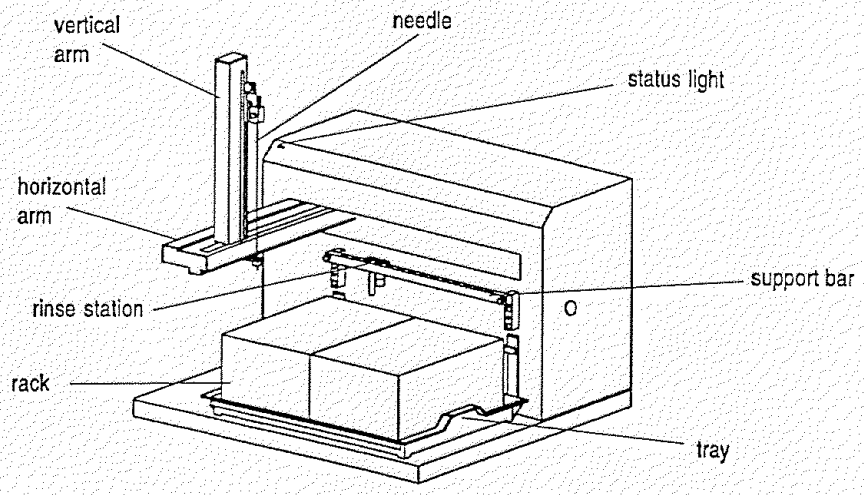
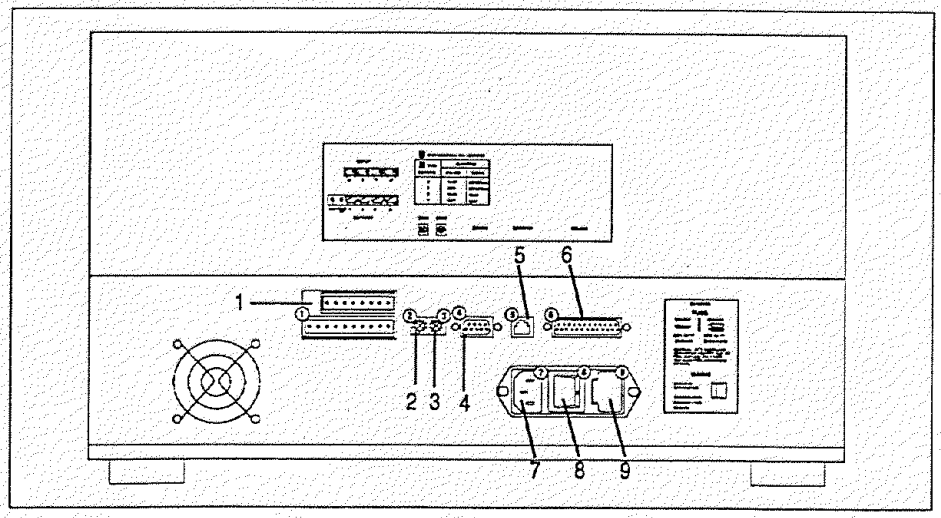


Figure 4-1 Possible Configuration of Autosampler

Figure 4-2 below illustrates the rear panel of the unit. Please note that the items with asterisks are not applicable to operating the autosampler with Phoenix 8000.



1. Input/Output (I/O) contact ports*
2. Unit ID selector* (default is 4)
3. Baud rate selector
4. Serial I/O channel port*
5. Keypad port*
6. RS-232 port
7. Power receptacle
8. Power switch
9. Fuse drawer

* These items are not applicable to operating the autosampler with Phoenix 8000.

Figure 4-2 Autosampler Rear Panel

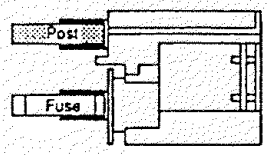
4.2 Tools and Supplies Needed

Before you begin assembling the autosampler, gather the following tools and supplies:

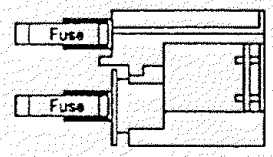
- Standard screwdriver
- Phillips head screwdriver
- Accessory packages shipped with unit.

4.3 Installing Fuses

Before operating the autosampler, you must first install fuses. *Figure 4-3* illustrates the two fuse drawers shipped with the unit.



Fuse installation for 100/120 voltage



Fuse installation for 220/240 voltage

Figure 4-3 Two Fuse Drawers Shipped With Unit

The following steps describe how to install fuses:

1. Locate the accessory package containing the fuse drawer appropriate for your line voltage.
2. Locate the accessory package containing the 2.0 amp fuses.
3. Install the fuse(s) into the fuse drawer. The fuse drawer for 100/120 V accepts one fuse. The fuse drawer for 220/240 V accepts two fuses.
4. Insert the fuse drawer into its receptacle in the back of the unit. See *Figure 4-2* for a rear panel diagram.

4.4 Connecting RS-232 Cable

The RS-232 port is used to transfer information between the autosampler and Phoenix 8000. For the location of the RS-232 port, refer to *Figure 4-2*.

Locate the RS-232 cable. Attach the male end of the cable to the port on the rear panel of the autosampler. Tighten the retaining screws. Attach the other end of the cable to the appropriate port on the rear panel of Phoenix 8000. Again, tighten the retaining screws.

4.5 Setting Baud Rate

The SW2 selector sets the baud rate for the unit. See *Figure 4-2* for location of the selector.

As a default, the baud rate selector is set to 0 for identifying a baud rate of 19200.

If your computer's baud rate is 9600, complete the following steps to change the setting for the SW2 selector to ~~1 of 3.~~ 4

1. Gently insert a small flat blade screwdriver into the SW2 selector on the rear panel and turn.
2. Align the white dot with ~~1 of 3.~~ 4

SW1 = 0

4.6 Removing Arm Locking Screw

During shipment, a screw locks the horizontal arm into place.

1. Locate and remove the black plastic plug located on the right side panel of the autosampler.
2. With your left hand, hold the horizontal arm into place.
3. Using a Phillips screwdriver, remove the arm locking screw, located inside the autosampler.
4. Insert the arm locking screw into its storage location on the rear panel.
5. Replace the plastic plug on the side panel.
6. Ensure that the horizontal arm can move by pushing it to the left as far as it will go.

Before packing the autosampler for shipment, always secure the horizontal arm using the arm locking screw.

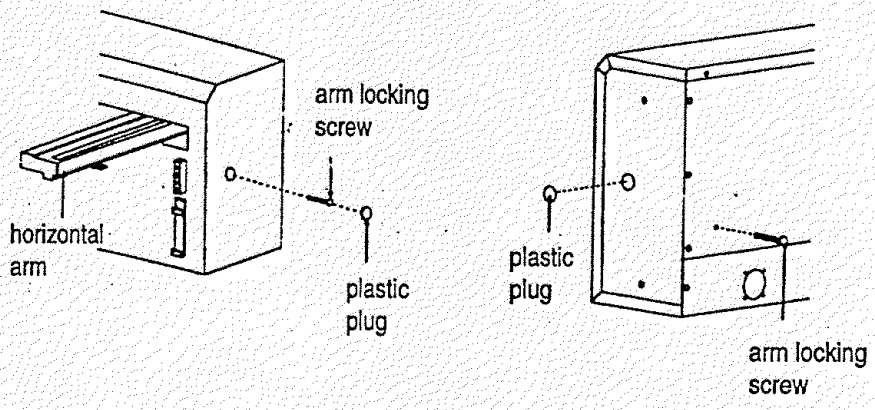


Figure 4-4 Arm Locking Screw Location

4.7 Installing the Vertical Arm

Before installing or changing the vertical arm, check that the power is turned off and the power cord is disconnected from the power socket. Also ensure that the arm locking screw has been removed and the horizontal arm can move.

To install the vertical arm:

1. Remove the cover plate from the front of the horizontal arm by removing its three screws.
2. Locate the hexagonal-shaped control rod and horizontal slider by looking down into the horizontal arm. See *Figure 4-5*.
3. Using your finger, press on the control rod where it passes through the horizontal slider. At the same time, pull the needle foot toward the front of the horizontal arm. This causes the white plastic plug and control rod to move forward slightly. When the white plastic plug is no longer flush with the front of the horizontal arm, remove it and the control rod from the horizontal arm.

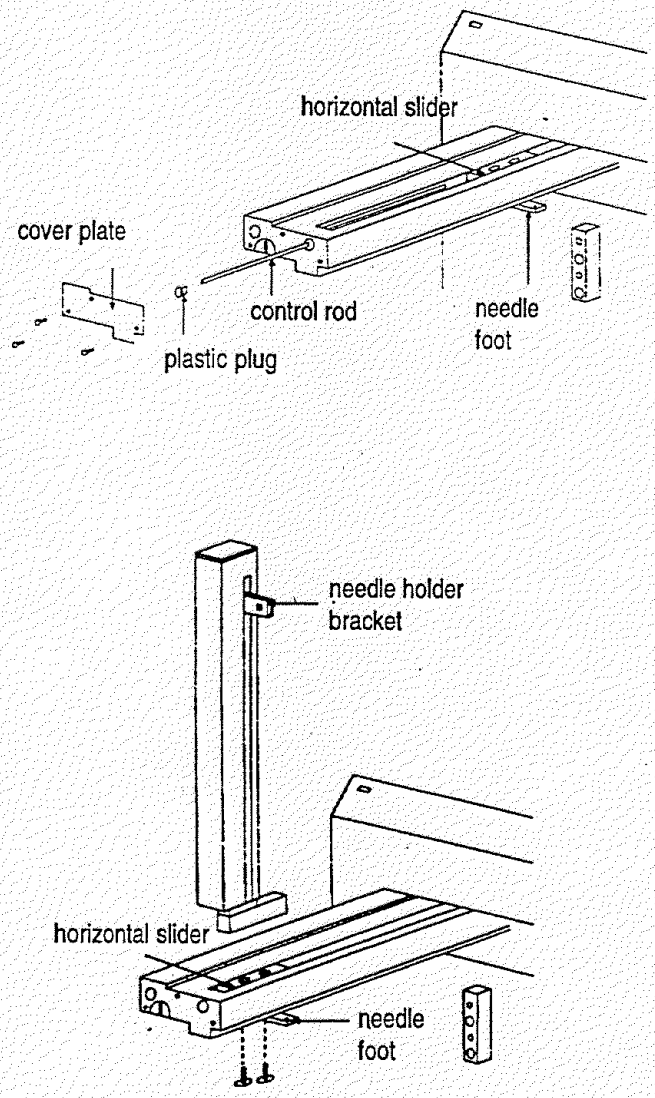


Figure 4-5 Installing the Vertical Arm

4. Pull the needle foot, toward the front of the horizontal arm, as far as it will go.
5. Position the vertical arm onto the horizontal slider. When viewed from the front of the autosampler, the vertical arm's needle holder bracket is on the right.
6. Use the supplied screws to secure the vertical arm to the horizontal slider. The screws insert into the bottom of the mounting holes in the horizontal slider. You may need to move the vertical arm back and forth slightly to align its mounting holes with those in the horizontal slider. (See *Figure 4-6*.)
7. Re-insert the control rod as far as it will go. While inserting the control rod, you may need to rotate it back and forth slightly to get it to pass through the gearing and motor drive socket in the horizontal slider.
8. Applying pressure at the vertical arm's base, push the vertical arm to the back of the horizontal arm.
9. While slightly moving the needle holder bracket up and down, push the control rod until it clicks into position.
10. Re-insert the white plastic plug.
11. Re-attach the cover plate to the front of the horizontal arm.

4.8 Installing the Needle

Locate the accessory packages containing the needle and the needle holder/ guide kit. When installing the needle, refer to *Figure 4-6* for placement of the needle, needle holder, and needle guide.

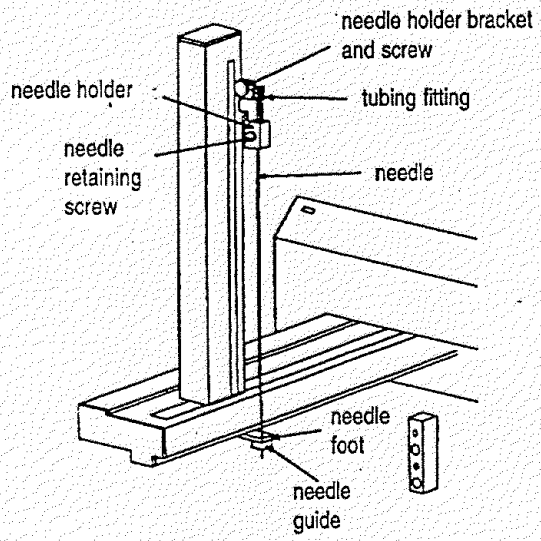


Figure 4-6 Installing the Needle

To install the needle guide:

1. Place the metal lock washer onto the needle guide.
2. Screw the needle guide with lock washer into place on the bottom side of the needle foot.

To install the needle holder, slide it into place on the needle holder bracket and secure with the supplied screw.

To install the needle:

1. Slide the needle into the tubing fitting.
2. Connect the transfer tubing to the tubing fitting.
3. Slide the needle assembly into the needle holder on the vertical arm making sure that the needle is centered over the needle guide.
4. Secure the needle by tightening the needle retaining screw.

4.9 Installing the Rinse Station and Support Bar

The support bar attaches to the front face of the autosampler. Its rear contains mounting holes for rinse stations and its top contains mounting holes for transfer ports and filler ports.

1. Attach the rinse station to the support bar. The default position for the rinse station is the left side of the support bar when the bar is attached to the autosampler. From left to right, the ports in a rinse station are the rinsing well, level-sensing rinsing well, and drain. See *Figure 4-7*. To attach tubing to the drain, locate the drain tubing. Remove the locking collar from the drain fitting. Slide the tubing through the locking collar. Attach the tubing to the drain fitting. Then screw the collar onto the drain fitting. Place the other end of the tubing in a drain receptacle, located lower than the tray.
2. Attach the support bar to the front of the autosampler using the knurled screws.

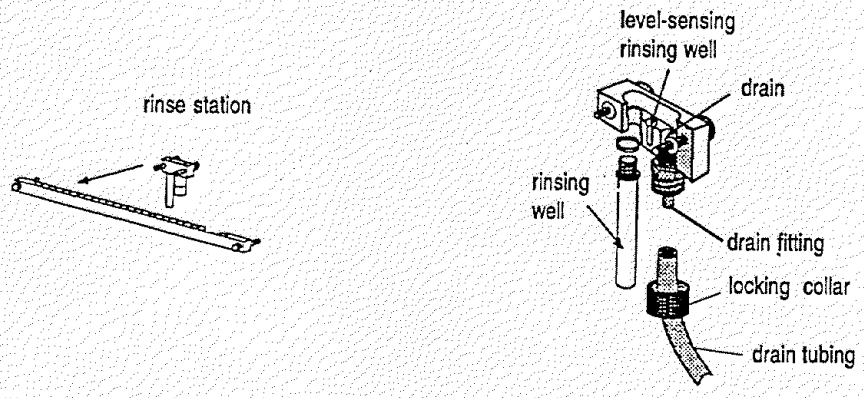


Figure 4-7 Installing the Rinse Station and Support Bar

NOTE: Depending upon your sight requirements, it may be necessary for you to attach a larger diameter piece of tubing over the existing drain tubing. To do this, cut existing drain tubing, leaving approximately 2-3" of drain tubing , then slide larger tubing over it.

4.10 Installing the Tray

The tray positions the racks and any accessories that fit onto the bed of the autosampler. It also contains liquids spills, such as those caused by overflowing vessels. The tray is installed in the lower position on the autosampler's bed.

Make sure that the tray fits securely and that the drain outlet is located at the left rear of the tray. Attach one end of the drain tubing to the drain outlet and place the other in a drain receptacle, located lower than the tray.

To remove the tray, first lift it straight up and then bring it back toward you.

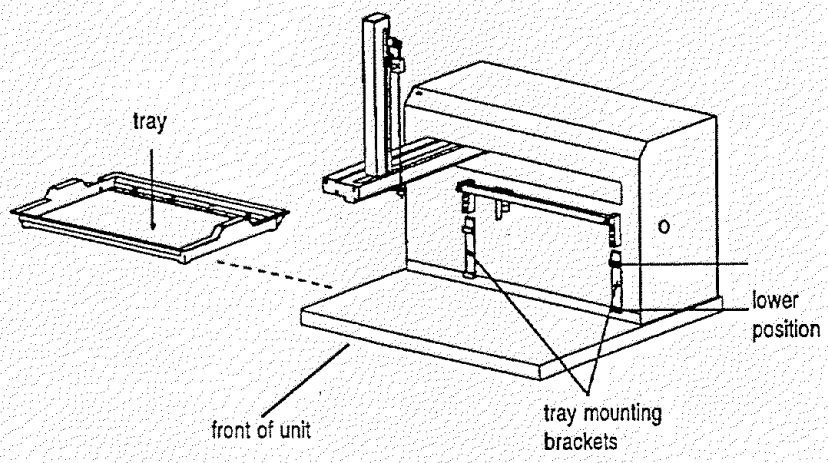


Figure 4-8 Installing the Tray

4.11 Installing the Rack

Place each rack into the tray so that the rack is perpendicular to the front panel of the autosampler. Make sure that the rack handles are positioned to front of the unit.

4.12 Connecting Power Supply

Locate the appropriate power cord for your line voltage.

Use the power cord to connect the sample changer to an AC power source. Surge protection is recommended.

5.1 Overview

Phoenix 8000 has four operating modes—Total Organic Carbon (TOC), Total Carbon (TC), Inorganic Carbon (IC), and Total Carbon Minus Inorganic Carbon (TC-IC).

This chapter discusses sample introduction and presents step-by-step instructions and flow diagrams for each of the four operating modes.

5.2 Sample Introduction

Before introducing a sample, Phoenix 8000 automatically rinses the syringe to eliminate any contaminants that may interfere with the testing process. This rinsing occurs through a loop sequence where the syringe is filled and discarded with the same sample you will be testing.

Because air bubbles in the syringe can affect test results, Phoenix 8000 automatically discards 0.5 ml from the top portion of the sample and 0.5 ml from the bottom portion of the sample (regardless of your sample size). For example, if you are testing a 1 ml sample, aspirate 2 ml into the syringe. Phoenix 8000 drains the first 0.5 ml to waste, delivers the next 1 ml to the reaction chamber, and drains the remaining 0.5 ml to waste. This method of sample introduction is referred to as "delivering" a sample. See *Figure 5-1*.

Note that the aforementioned method of delivering a sample is common to all the operating modes and is not illustrated in the flow diagrams.

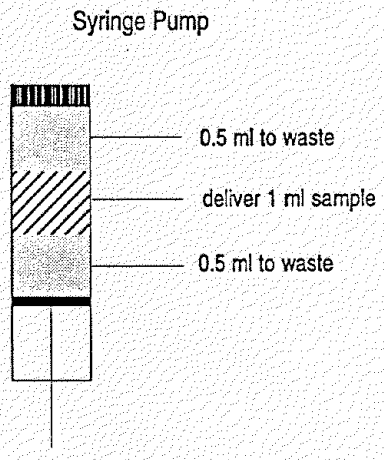
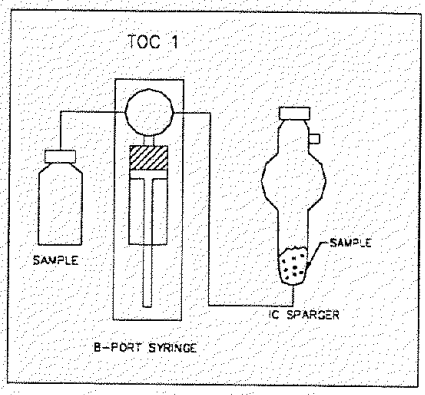


Figure 5-1 Delivering a Sample

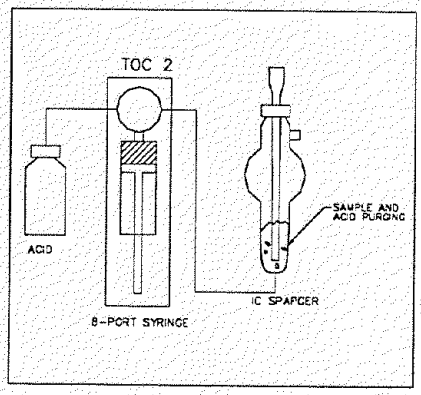
5.3 Total Organic Carbon (TOC)

In the TOC mode, inorganic carbon is removed by acidification and sparging. The remaining carbon in the sample is measured as TOC. The flow for this mode is as follows:

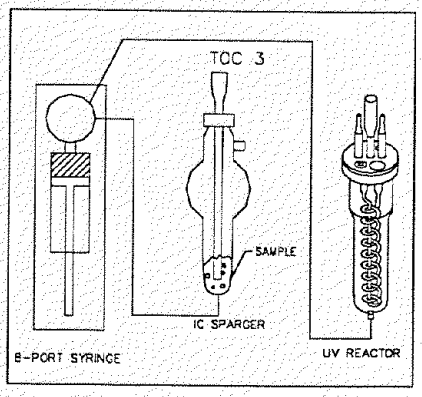
- Syringe is rinsed with sample
- Sample is introduced into syringe
- Sample is delivered to IC sparger



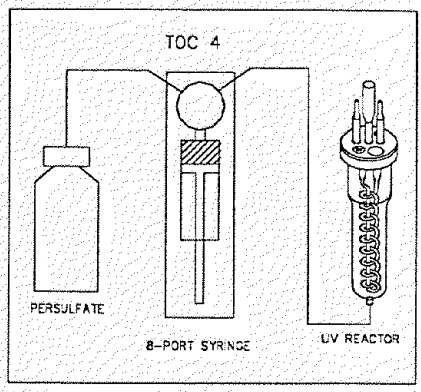
- Syringe is rinsed with water
- Acid is introduced into syringe
- Acid is delivered to IC sparger



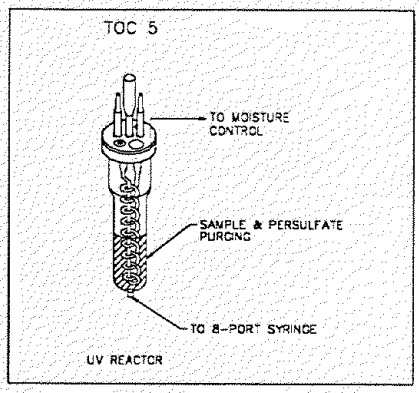
- Carrier gas is sent to IC sparger to purge sample of inorganic carbon to vent
- Sample in IC sparger is used to rinse syringe
- Treated sample is transferred to UV reactor via syringe
- Syringe is rinsed with water



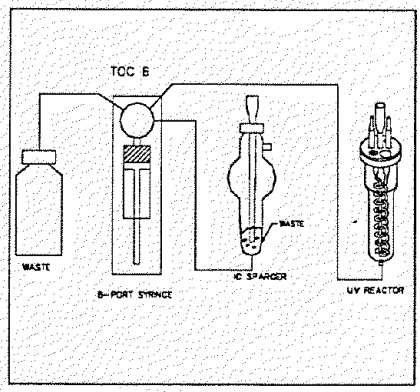
- Persulfate is delivered to UV reactor
- DI water is delivered to UV reactor to fill UV chamber (if needed)

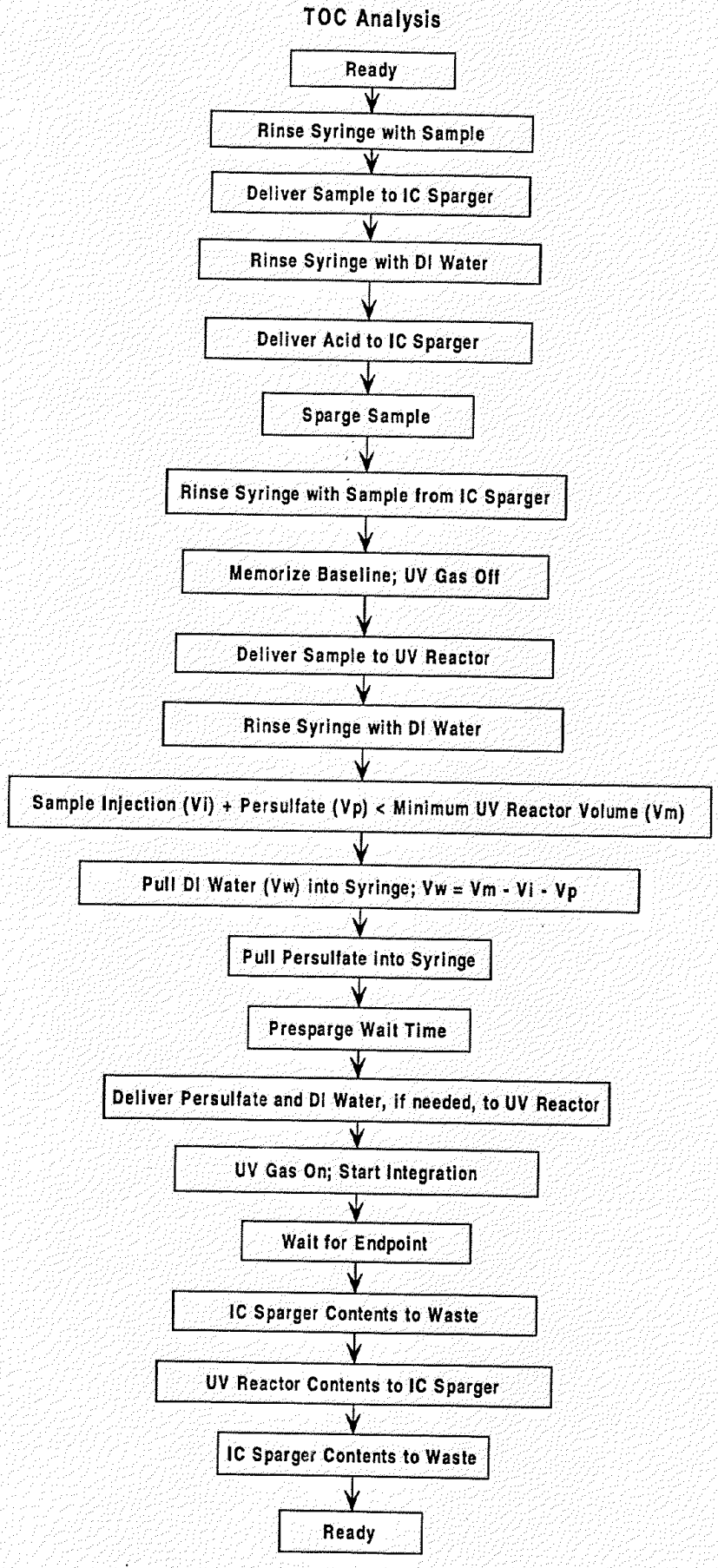


- Carrier gas is swept through UV reactor to carry CO_2 out of the UV chamber
- CO_2 is swept through the moisture removal system
- CO_2 is swept through halogen scrubber



- CO_2 is swept to NDIR to measure level of carbon
- NDIR signal is linearized and sent to computer to analyze
- IC sparger is emptied to waste
- UV reactor is emptied to waste
- IC sparger is rinsed with water

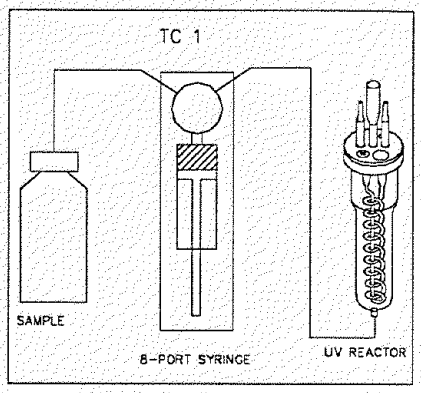



TOC Analysis
Ready
Rinse Syringe with Sample
Deliver Sample to IC Sparger
Rinse Syringe with DI Water
Deliver Acid to IC Sparger
Sparge Sample
Rinse Syringe with Sample from IC Sparger
Memorize Baseline; UV Gas Off
Deliver Sample to UV Reactor
Rinse Syringe with DI Water
Sample Injection (Vi) + Persulfate (Vp) < Minimum UV Reactor Volume (Vm)
Pull DI Water (Vw) into Syringe; Vw = Vm - Vi - Vp
Pull Persulfate into Syringe
Presparge Wait Time
Deliver Persulfate and DI Water, if needed, to UV Reactor
UV Gas On; Start Integration
Wait for Endpoint
IC Sparger Contents to Waste
UV Reactor Contents to IC Sparger
IC Sparger Contents to Waste
Ready

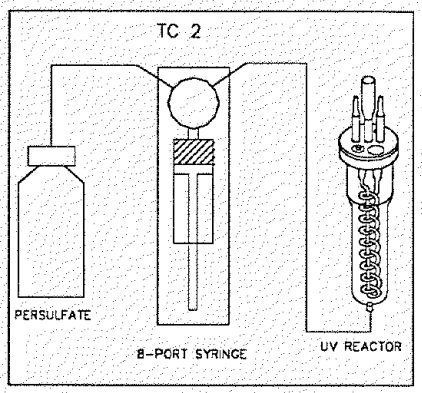
5.5 Total Carbon (TC)

TC is the measurement of all the carbon in the sample, both inorganic and organic, as a single parameter. The flow for this mode is as follows:

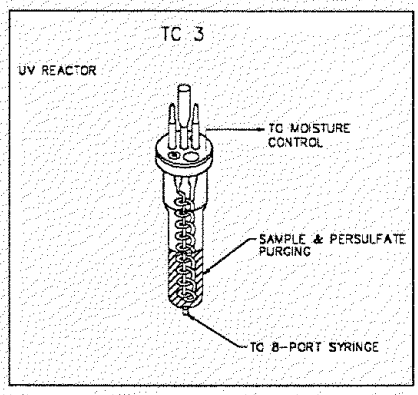
- Syringe is rinsed with sample
- Sample is introduced into syringe
- Sample is delivered to UV reactor



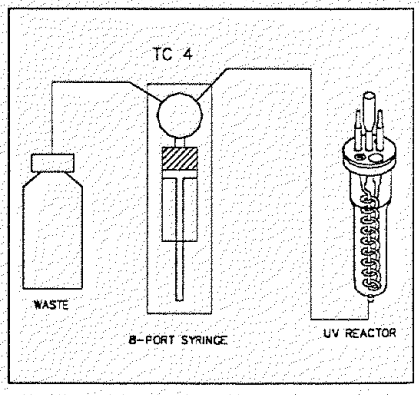
- Syringe is rinsed with water
- Persulfate/acid mixture is introduced into syringe and delivered to UV reactor
- DI water is delivered to UV reactor to fill UV chamber (if needed)



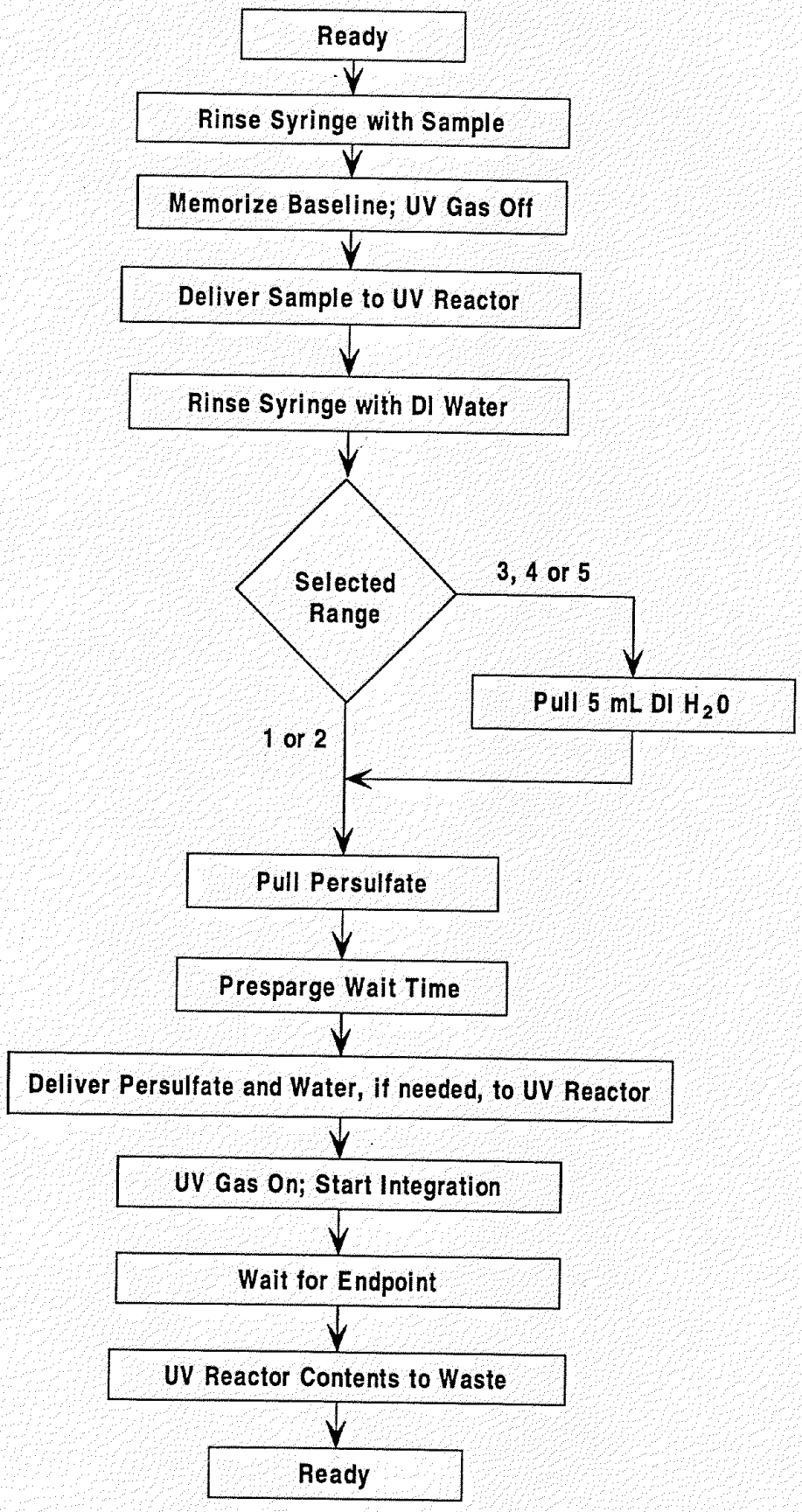
- Nitrogen is swept through UV reactor to carry CO_2 out of UV reactor
- CO_2 is swept through the moisture removal system
- CO_2 is swept through halogen scrubber



- CO_2 is swept to NDIR to measure level of carbon
- NDIR signal is linearized and sent to computer to analyze
- UV reactor is emptied to waste

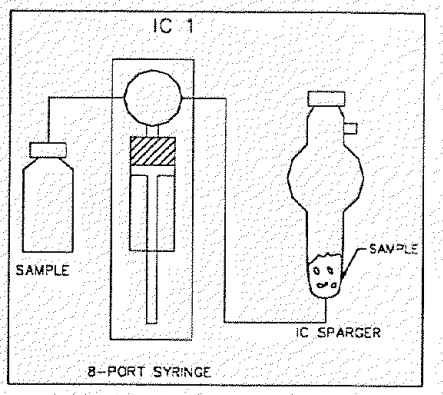


TC Analysis

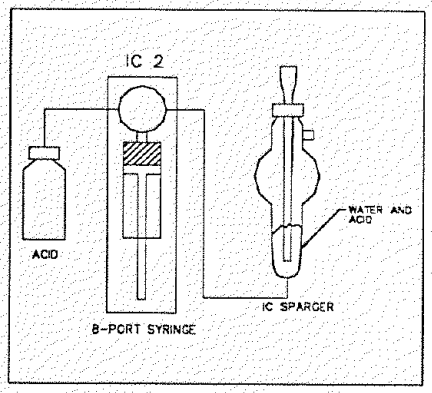

Ready
Rinse Syringe with Sample
Memorize Baseline; UV Gas Off
Deliver Sample to UV Reactor
Rinse Syringe with DI Water
Selected Range
3, 4 or 5
Pull 5 mL DI H_2O
1 or 2
Pull Persulfate
Presparge Wait Time
Deliver Persulfate and Water, if needed, to UV Reactor
UV Gas On; Start Integration
Wait for Endpoint
UV Reactor Contents to Waste
Ready

5.6 Inorganic Carbon (IC)

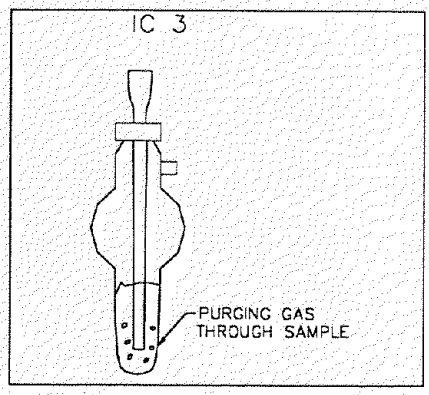
IC is analyzed in liquid samples by acidifying with an inorganic acid to pH 3 or lower and sparging with inert gas. The flow for this mode is as follows:



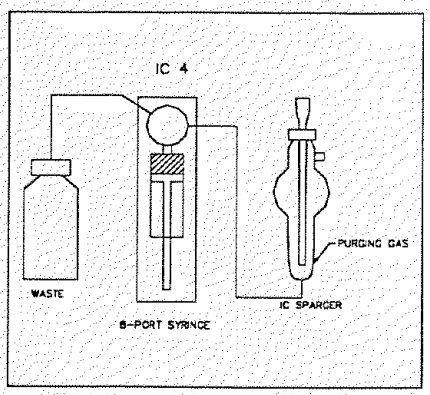
- Syringe is rinsed with sample
- Sample is introduced into syringe
- Sample is delivered to IC sparger



- Syringe is rinsed with water
- Acid is introduced into syringe
- Acid is delivered to IC sparger
- DI water is delivered to IC sparger to fill IC chamber (if needed)

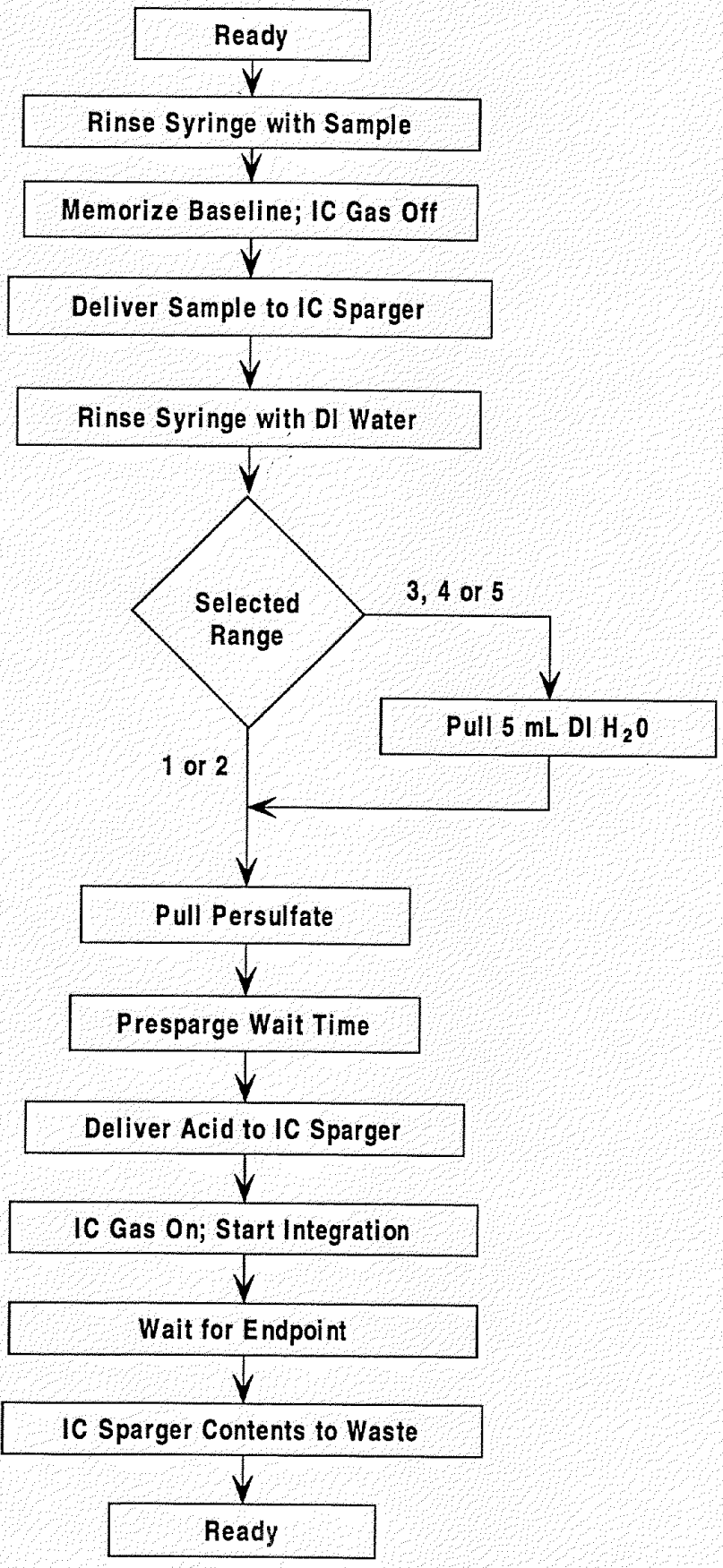


- Carrier gas is swept through IC chamber to carry CO_2 out of IC sparger
- CO_2 is swept through moisture removal system
- CO_2 is swept through halogen scrubber



- CO_2 is swept to NDIR to measure level of carbon
- NDIR signal is linearized and sent to computer to analyze
- IC sparger is emptied to waste

IC Analysis


Ready
Rinse Syringe with Sample
Memorize Baseline; IC Gas Off
Deliver Sample to IC Sparger
Rinse Syringe with DI Water
Selected Range
3, 4 or 5
Pull 5 mL DI H_2O
1 or 2
Pull Persulfate
Presparge Wait Time
Deliver Acid to IC Sparger
IC Gas On; Start Integration
Wait for Endpoint
IC Sparger Contents to Waste
Ready

5.7 Total Carbon Minus Inorganic Carbon (TC-IC)

The TC-IC mode is also referred to as TOC measurement by difference. This approach requires two analyses: one to measure TC, and one to measure IC. (See sections 5.5 and 5.6 for flow diagrams of these modes.) The difference between these two measurements is rigorously TOC.

6.1 Overview

This chapter discusses installing **TOC Talk**, the Phoenix 8000 software (p/n 14-7186-075), setting flow rates and pressure, calibration, and running the analyzer.

6.2 Installing the TOC Talk Software

User interface is achieved through TOC Talk. The Tekmar-Dohrmann Setup program properly installs the software on your computer. The TOC Talk program uses roughly 5 MB hard disk space and data files use an average of 20 MB of disk space per month. If you are installing a newer version of TOC Talk over an older version, you will need to manually delete the old icon(s) and program group(s) before installing the newer version. You do not need to delete any old program files. If an older copy exists, you may want to backup its directory along with its subdirectories to access these files at a later date.

NOTE: Refer to your Windows user manual for help with backing up your directory to a new location.

The following steps describe how to install TOC Talk:

To Install TOC Talk in Windows 3.x:

1. Start **Windows 3.1** or higher.
2. In **Program Manager**, choose **Run** from the **File** menu.
3. Place Disk 1 in a floppy drive. In the Run dialog box, type the letter of the drive and setup (for example, A:\setup), then click the **OK** button.
4. The first Setup window appears.
5. Follow the instructions on the screen.

After installation, the following icons appear in the TOC Talk program group:

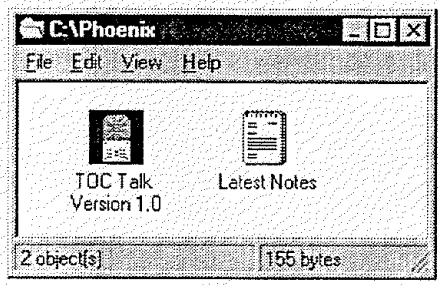


Figure 6-1 TOC Talk Program Group

To Install TOC Talk in Windows 95:

1. Start **Windows 95.**
2. Click on **Start** and choose **Run.**
3. Place Disk 1 in a floppy drive. In the **Open** dialog box, type the letter of the drive and setup (for example, A:\setup), then click the **OK** button.
4. The first Setup window appears.
5. Follow the instructions on the screen.
6. When installation is complete, the **TOC Talk icon** (see above) will be placed in the Programs group.
7. The **Latest Notes** icon permits the user to read current software information. Double click on this icon to read this text document.

6.3 Setting Flow Rates and Pressure

Once the software is installed and the system is turned on, it is critical to set the proper flow rate before beginning analyses. This section describes how to establish proper flow rates through Phoenix 8000.

Before beginning the steps to adjust flow rate, verify the following:

1. The main pressure in the gas supply tank should not be below 500 psi. If you have a psi reading of 500 or lower, change the tank.

WARNING!

Pressure to Phoenix should never exceed 35 psi after regulator.

2. For optimal performance, Phoenix 8000 should have a dedicated gas supply.
3. The length of 1/8" tubing from the gas supply tank should be no longer than five feet from the regulator to Phoenix 8000.

NOTE: If you must share a gas supply tank with another instrument, or if you must place Phoenix 8000 farther than five feet from the gas supply tank, place a regulator and gauge next to the unit.

You are now ready to set the flow rate:

1. After you have verified the above, turn on the gas supply to Phoenix 8000 and dial in 30 psi on the regulator.
2. Turn **On** the power switch located on the back pael of the phoenix 8000 unit.
3. Launch **TOC Talk** software.
4. If initialization with the Phoenix 8000 is not made, select the correct communications port from the **Setup--Communications** menu. If proper connection is made, the Phoenix 8000 will re-initialize.
5. Choose **Setup** from the main screen.
6. From **Setup** choose Instrument. The following screen appears.
7. Select the System--Ready mode.

NOTE: Step 7 is necessary to get reading in step 10.

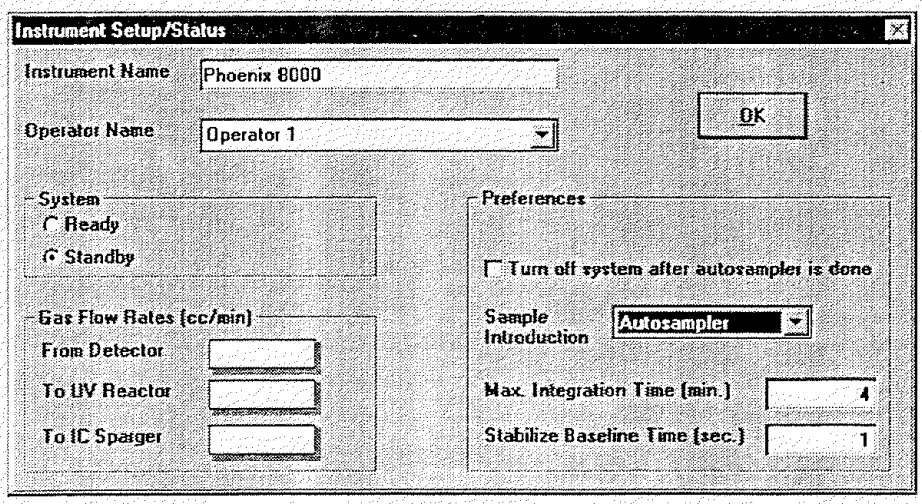


Figure 6-2 Instrument Setup Screen

8. Calibrate flowmeters (See page 6-22 *Diagnostics - Flowmeter Calibration.*)
9. Under **Gas Flow Rates (cc/min),** locate the reading for **From Detector.**
10. The recommended flow rate for Phoenix 8000 is 200 cc/min (±10%). Adjust the pressure from the gas supply tank regulator until the "From detector" reading reaches 200 cc/min.
11. If you purchased the optional UV flowmeter or IC flowmeter, you can check the flow rates of those components on this screen also. They should have the same reading as the flow rate for "From detector."

    If the optional flowmeters are not installed, the "To UV reactor" and "To IC sparger" lines will be grayed out and you must perform a manual reading for flow rates of these components. (See Chapter 7.)
12. If you cannot maintain a 200 cc/min reading, or if you must go above 35 psi from the gas regulator to achieve this reading, you must perform a system leak check. (See Chapter 7.)

WARNING!

Do not set gas pressure above 35psi. Doing so may cause damage to your unit. If you find that you need to increase the pressure above 35psi, you most likely have a gas leak.

6.4 Understanding Calibration

Before analyses, Phoenix 8000 must be calibrated. A response factor correlates the raw counts of the instrument to a **known** amount of carbon in a sample, or what is referred to as a **standard.** Standards are made by adding carbon to ultrapure water to achieve determined levels of carbon. It is important to use the same source of water for all calibration standards because the carbon in the preparation water is part of the calibration curve.

Standard analyses have, in general, 3 major sources of carbon content: measured carbon content added to the ultrapure water, carbon from the preparation water itself, and carbon associated with the reagents used to perform the relevant analysis. When we calibrate the instrument to a y=mx+b linear fit, the constant carbon contribution of the prep water and the reagent are represented by the y-intercept.

$$y = mx + b_{cal}$$

$$b_{cal} = b_{reagent} + b_{rinse\ water}$$

The slope of the curve, m, represents the relationship of the measured carbon in the standard to the response of the instrument since only this source of carbon changes between any set of standards.

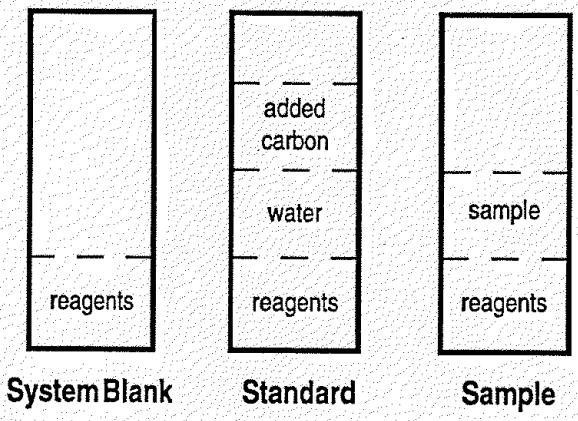
NOTE: Running your preparation water as a "zero" standard can be an excellent way to calibrate the low end of the concentration range for any method. (See *Calibration - Standards* for more information)

After calibration, the slope m will be used as our response factor in sample analysis. However, the y-intercept, b_{cal}, will NOT be used because the carbon associated with a standard's preparation water, $b_{rinse\ water}$, is not present in the sample. The linear equation for samples is simply:

$$y = mx + b_{reagent}$$

where $b_{reagent}$ is the relevant system blank which is measured separately.

Sometimes the user will want to verify their calibration curve. However, if a standard is run as a sample, the final result will be off by the carbon contribution of the preparation water. In these cases, the user must run the standard as a **calibration verification** instead of sample. This type of analysis will apply the correct y-intercept, b_{cal}, to the analysis.

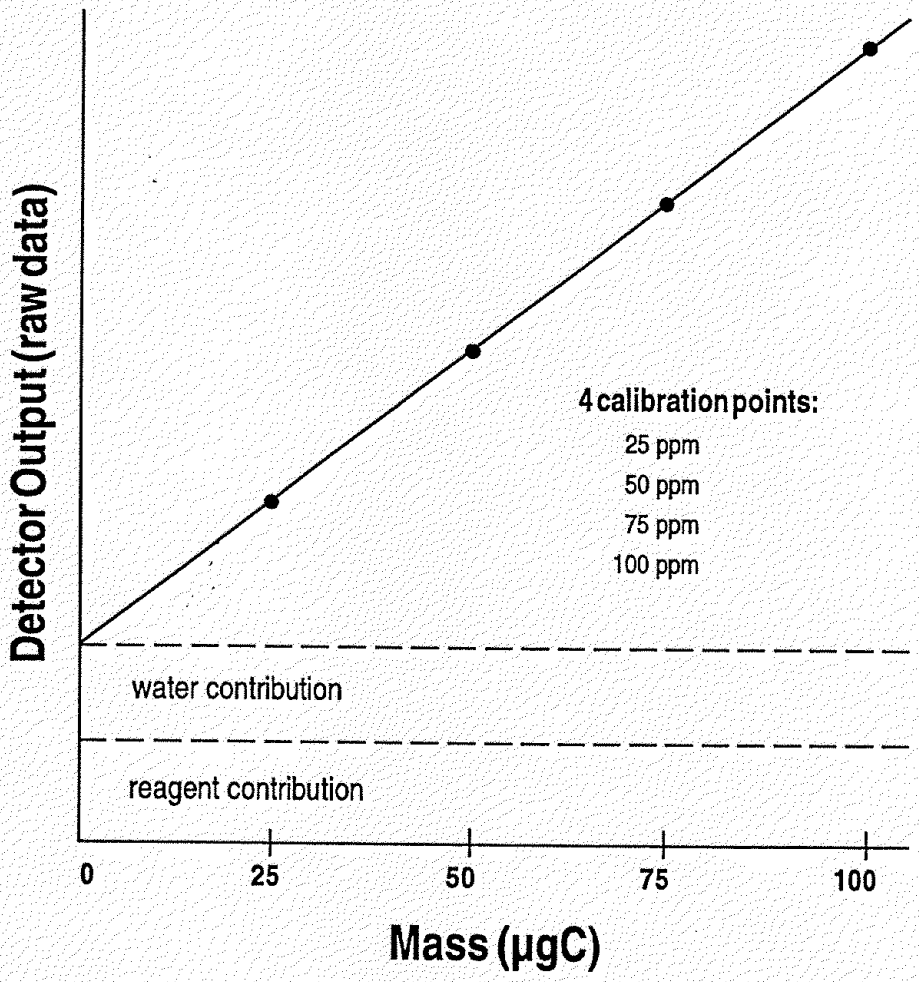


$$\text{"Blank"}\ \text{Reagent} = C_{(Reagent)}$$

$$\text{Standard} = C_{(Reagent)} + C_{(Water)} + C_{(Standard)}$$

$$\text{Sample} = C_{(Reagent)} + C_{(Sample)}$$

Figure 6-3 Reagent, Standard, and Sample Composition



Slope = calibration factor
Intercept = calibration blank

Figure 6-4 Calibration Slope

NOTE: Proper calibration is essential because the data read is unique to your NDIR detector. Proper calibration setup allows the software to accurately interpret data. You must send a predetermined amount of µgC through the detector in order to set up the calibration curve for the software to analyze.

6.5 Using TOC Talk

This section covers the basic operations of Windows software.

Use your mouse and PC keyboard to fill in parameters and choose options.

Using the Mouse

Choose menus, options, and buttons with a click of your mouse button. Menus are at the top of the screens. They contain more options for using TOC Talk.

Options are denoted by radio buttons and check boxes. When you choose an option by clicking one of the radio buttons, a black dot appears in the middle of the button. When you choose an option by clicking one of the check boxes, a check mark appears in the middle of the box.

Buttons have different functions depending on the name of the button. Click the button to choose it.

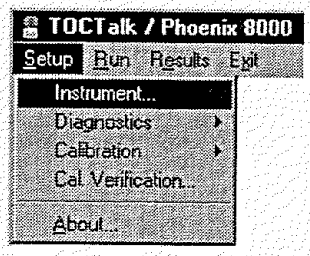
Using the Keyboard

Keep the following keys in mind for use with TOC Talk:

Tab	moves to the next option, button, entry
Shift + Tab	moves to the previous option, button, entry
Shift	chooses the upper character on a key
Ctrl	and/or **Alt** used with another key, allows you to use the keyboard as a shortcut to choosing items and options, rather than using the mouse
Enter	accepts the screen's parameters
Delete	erases an entire entry

Using the Menus

Press **Alt** and the underlined letter to pull down the menu OR move the mouse until the arrow is on the menu name. Click once to pull down the menu.



Example
Press Alt + S to pull down the File menu or click on it once with the mouse. To choose a menu item, press the underlined letter (I, D, C, L, A) or click on the item with the mouse. If you have not already opened the menu, you may choose one of the keyboard shortcuts listed next to the menu item.

Figure 6-5 Example of Using Keyboard Shortcuts

Press the shortcut key or key combinations to choose the menu option. These shortcuts work only if you have already pulled down the menu down. After the menu is opened, only press the underlined letter *or* move the mouse until the arrow is over the option. Click once to choose the menu option.

When you double-click on the **TOC Talk program icon**, the TOC Talk **Control screen** appears.

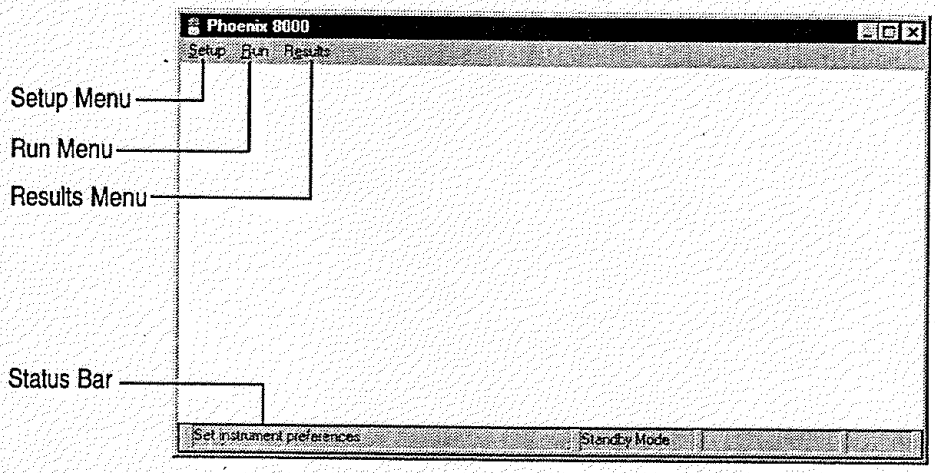


Figure 6-6 TOC Talk Control Screen

The **Control screen** contains menus with keyboard shortcuts, setup options, the run option, results, and a status bar that depicts event list items in real time along with mode status and current NDIR mV output.

6.6 Instrument Setup

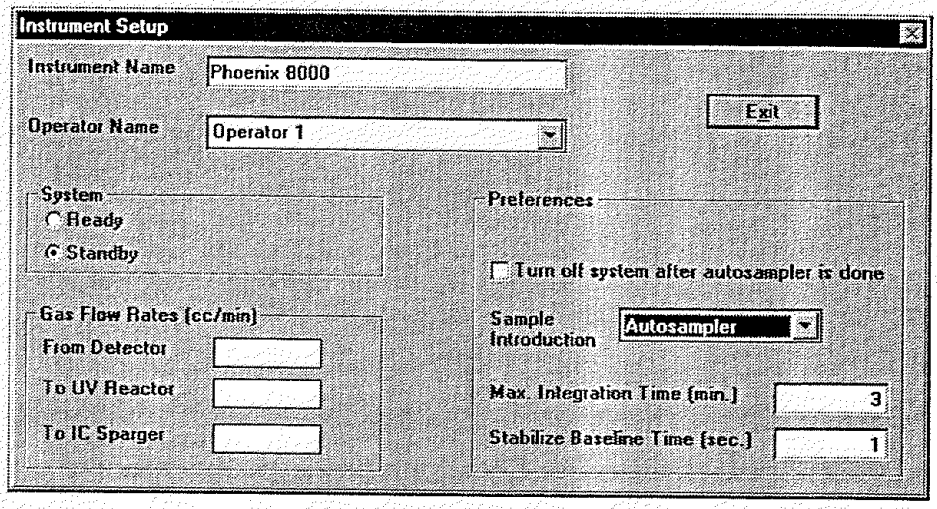


Figure 6-7 Instrument Setup Screen

System

Ready

Ready Mode turns on the UV Lamp, Main Gas Inlet Valve, Gas To UV Chamber, and routes carrier gas from the IC Sparger to vent and from the UV Reactor to the NDIR. User must be in Ready Mode to execute an analysis.

Standby

Standby Mode shuts all components off with the exception of the NDIR. Shutting off the NDIR will incur a minimum two hour stabilization delay upon startup. Tekmar-Dohrmann recommends that the Phoenix 8000 be in Standby Mode when not in use.

Gas Flow Rates (cc/min)

Displays flow rates for the relevant selection. See *Flowmeter Calibration* to calibrate flowmeters.

From Detector (Standard Flowmeter)

Detects flow after the NDIR. This flowmeter can be calibrated manually from the gas outlet port at the rear of the instrument.

To UV Reactor (Optional Flowmeter)

Detects flow entering the UV Reactor. This flow can be calibrated manually from the carrier gas line (1/8" blue striped) entering the fritted sparger at the top of the UV Reactor.

To IC Sparger (Optional Flowmeter)

Detects flow entering the IC Sparger. This flow can be calibrated manually from the carrier gas line (1/8" red striped) entering the fritted sparger at top of the IC Sparger.

Preferences

Turn off System after Autosampler is Done

Selecting this feature will allow the unit to automatically go to Standby Mode after an Autosampler run.

Sample Introduction

Configures system for either Automatic Syringe (manual operation) or Autosampler operation using the pull down menu.

WARNING!

Do not change Sample Introduction type in the middle of an analysis. Doing so can lead to erroneous data and damage to the instrument.

Max. Integration Time (min.)

Time allotted for the reaction portion of an analysis to take place. Default is set to 4 minutes.

Stabilize Baseline Time (sec.)

Some Analysis Modes such as TC, IC, TC-IC may require added wait between analyses to allow proper return of the baseline to a stable value. This is not usually necessary for TOC mode.

6.7 Diagnostics-Valves

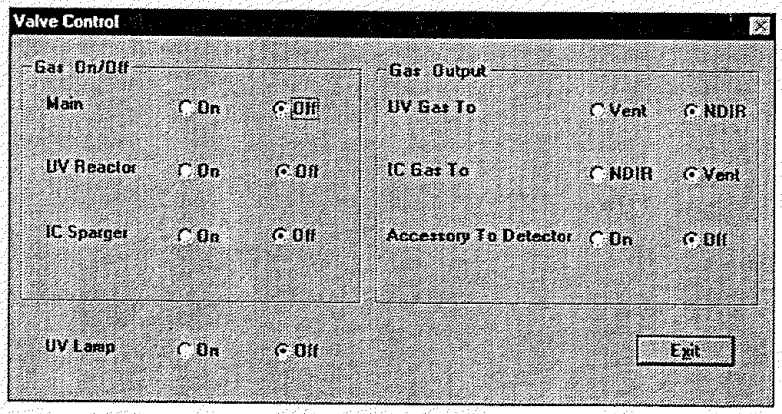


Figure 6-8 Diagnostic - Valves Screen

WARNING!

Altering this screen during an analysis can cause erroneous data and damage to the instrument.

Gas On/Off

Main

Primary carrier gas regulator. This valve must be on in order for carrier gas to flow to all other parts of the instrument with the exception of the NDIR purge gas (20 cc/min).

UV Reactor

Carrier gas valve for the UV Reactor.

IC Sparger

Carrier gas valve for the IC Sparger

Gas Output

UV Gas To

Valve switch for sample gas from the UV Reactor to the NDIR or Vent.

IC Gas To

Valve switch for sample gas from the IC Sparger to the NDIR or Vent.

Accessory To Detector (Optional)

Valve switch for sample gas from an accessory to the NDIR or Vent.

UV Lamp

Controls power to the UV Lamp.

6.8 Diagnostics - Syringe

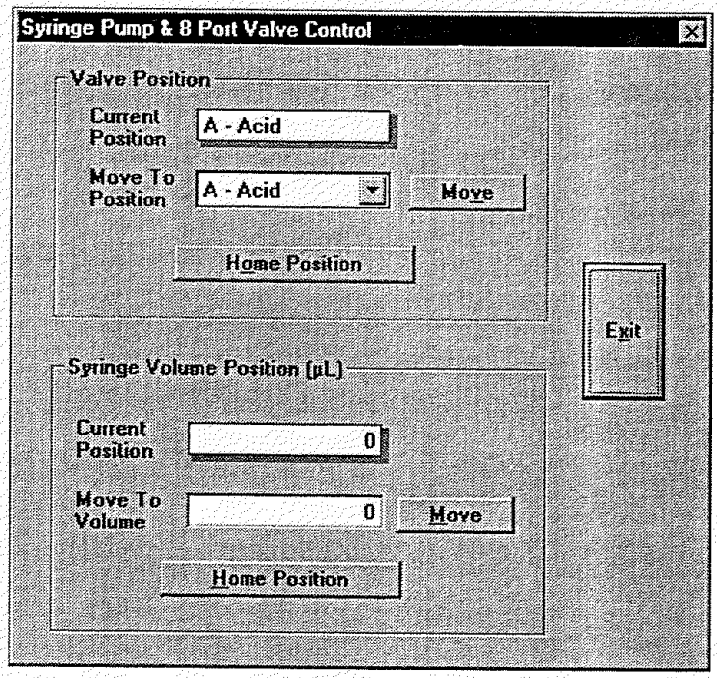


Figure 6-9 Diagnostics - Syringe Screen

WARNING!

Altering this screen during an analysis can cause erroneous data and damage to the instrument.

Valve Position

This section of the syringe diagnostic deals with the operation of the 8-port valve on the syringe pumper.

Current Position

Displays current position of the outer port of the 8-port valve. The common port is always connected to the syringe.

Move To Position

User can choose the outer port position here by clicking on the pull down window or the display box and then clicking on the **Move** button.

Home Button

Automatically switches the outer port of the 8-port valve to position **A-Acid.**

Syringe Volume Position (µL)

This section of the syringe diagnostic deals with the position of the syringe on the syringe pump.

Current Position

Displays current position of the syringe in microliters.

Move To Volume

User can move the syringe by dialing in the absolute position of the syringe in microliters and clicking the **Move** button.

Example: If the user want to pull 10000 µL from a syringe already located at 2000 µL then the user would dial in 12000µL and click the **Move** button.

Home Button

Automatically moves the syringe to 0 µL.

6.9 Diagnostics-AutoSampler

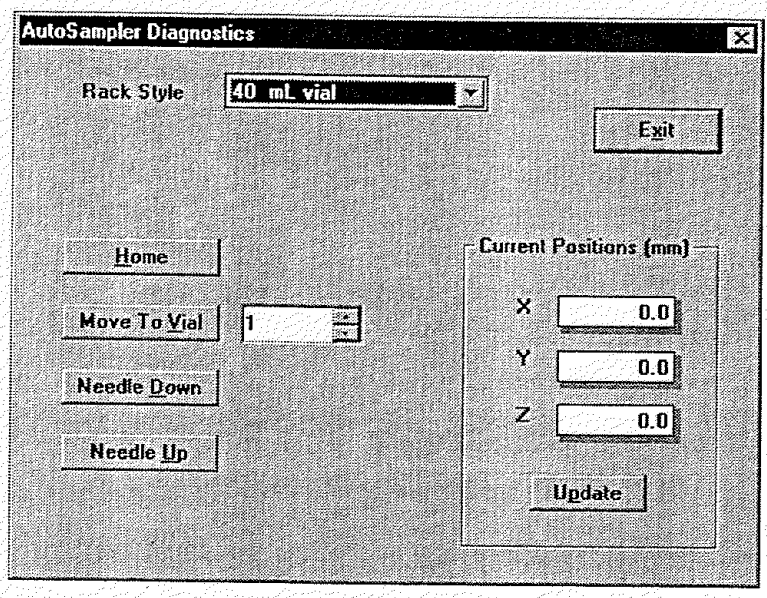


Figure 6-10 Autosampler Diagnostic Screen

WARNING!
Altering this screen during an analysis can cause erroneous data and damage to the instrument.

Rack Style

Select the rack style you are using by clicking the pull down menu or the display box.

Home

Click this button to home the autosampler

Move To Vial

Click this button to move the autosampler needle to the position selected in the display box to the right of the button. The position number can be changed by clicking the arrow bar or the display box.

NOTE: Special Position Numbers
1001 thru 1014 - support bar ports
1015 - Rinse Station

Needle Down

Moves autosampler needle down

Needle Up

Moves autosampler needle up

Current Positions (mm)

This section displays the current position, in millimeters, of the autosampler in the X, Y, and Z planes when the **Update** button is activated.

6.10 Diagnostics-Communications

This section allows the user to switch the communications port or re-establish communications when necessary.

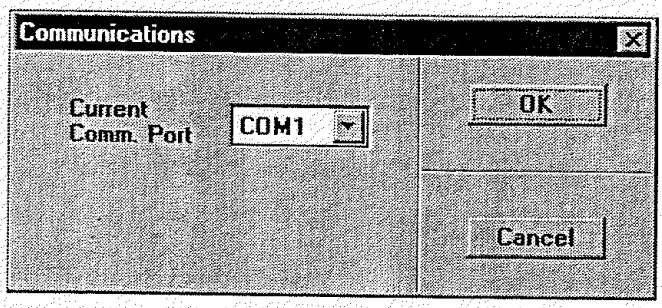


Figure 6-11 Diagnostics-Communications Screen

Comm. Port

Select the Communication port from the PC to the Phoenix 8000 here by clicking the pull menu or the display box.

OK Button

Clicking this button accepts the communications port selection and re-initializes the Phoenix 8000.

WARNING!

Clicking the OK button will comprimise the date of current run.

Cancel Button

Clicking this button exits the communications window without re-assigning the port and without re-initializing the Phoenix 8000.

6.11 Diagnostics-Flowmeter Calibration

In thi
instru

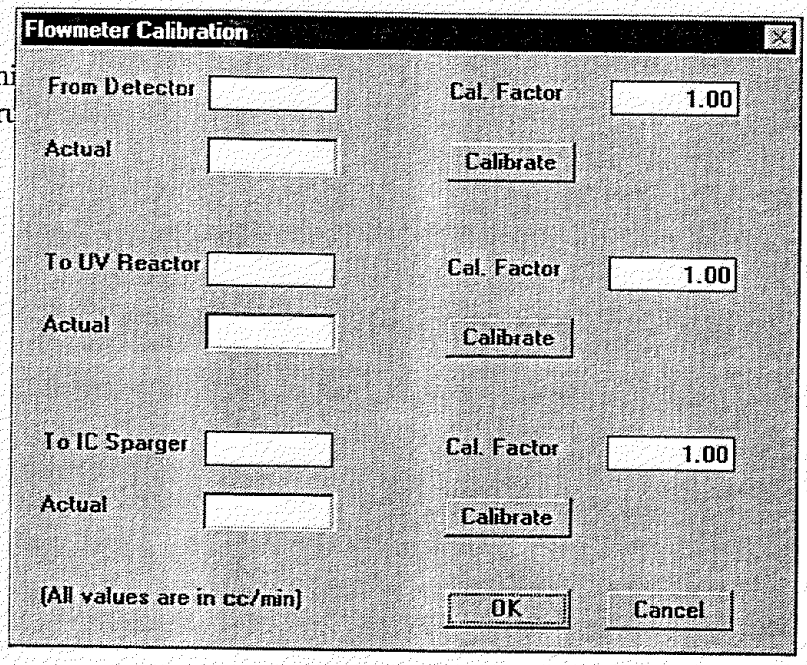


Figure 6-12 Flowmeter Calibration Screen

Calibrating a Flowmeter

To calibrate a flowmeter, the user must first measure the relevant flow with an independent flowmeter.

WARNING:
Make sure the UV Lamp is off when calibrating the optional UV Reactor or IC Sparger flowmeters.

The best place on the instrument to measure this flow is:

From Detector

This flowmeter can be calibrated manually from the gas outlet port at the rear of the instrument. Make sure that only sample gas from the UV Reactor is going to the detector. (See section 6.8, "Diagnostic-Valves")

To UV Reactor (Optional)

This flow can be calibrated manually from the carrier gas line (1/8" blue striped) entering the fritted sparger at the top of the UV Reactor. Make

sure both the Main and UV Reactor gas valves are **ON**. (See section 6.8, "Diagnostic-Valves")

To IC Sparger (Optional)

This flow can be calibrated manually from the carrier gas line (1/8" red striped) entering the fritted sparger at top of the IC Sparger. Make sure both the Main and IC Sparger gas valves are **ON.** (See section 6.8, "Diagnostic-Valves")

6.12 Calibration - Standards

Once the actual flow for a specific flowmeter is known, enter it, in units of cc/min, in the **Actual** display box. Reconnect the gas lines, if necessary, and if the flowmeter reading is stable, press the **Calibrate** button. The ratio of Actual flow to raw flow will be displayed in the **Cal. Factor** display box.

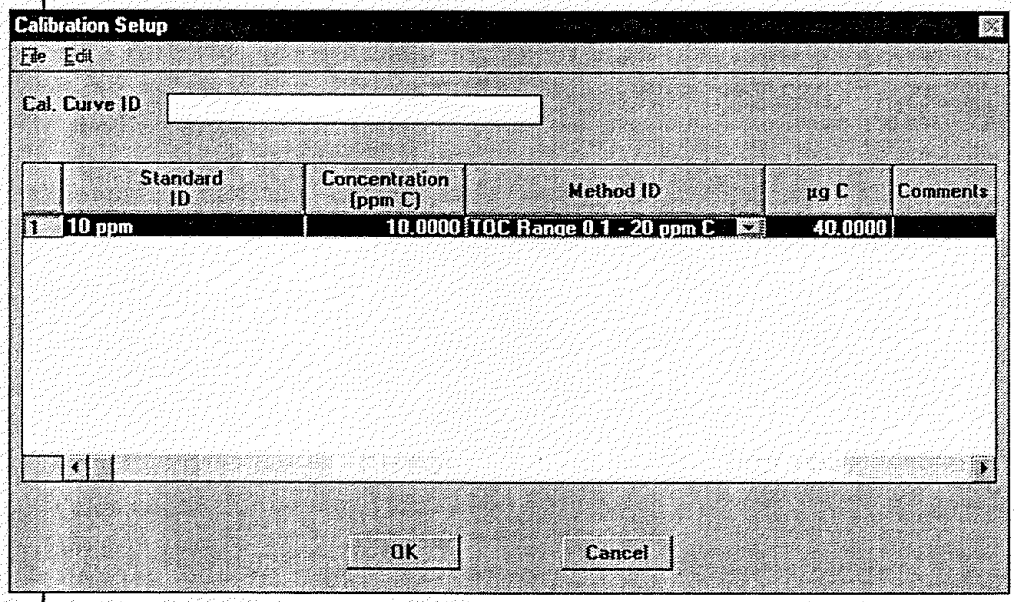


Figure 6-13 Standard Calibration Setup Screen

Shortcut Key

User can get more rows by pressing the down arrow on the PC keyboard.

Cal. Curve ID

User defined identification for a specific calibration curve; upon saving, may contain up to 32 characters.

Standard ID

User can define the identification for specific standards to be used in the calibration curve; may contain up to 32 characters.

Concentration (ppm C)

User must enter the concentration of the standard, in parts per million carbon, here.

Note: Software will not accept 0 as a concentration. When running preparation water as a standard, user may enter an insignificant decimal (such as 0.00001) to approximate 0.

Method ID

User must choose the method that the standard will reference when it is being analyzed.

WARNING!

DO NOT run different methods for the same calibration curve. Doing so will cause the amount of carbon in the preparation water and reagents to vary and adversely affect the slope of the curve or calibration factor. (See *6-4 Understanding Calibration*).

WARNING!

DO NOT choose methods which are not meant for sample analysis such as the sample blanks or cleaning procedure.

NOTE: Tekmar-Dohrmann recommends calibrating on 0.1 - 20 ppm method for sample runs between 2 ppb - 100 ppb, 0.1 ppm - 20 ppm methods for sample runs between 0.1 ppm - 20 ppm, and 20 ppm - 200 ppm methods for sample runs between 20 ppm - 10000 ppm. 200 ppm - 1000 ppm and 1000 ppm - 10000 ppm methods are automatic dilution methods that introduce the same range of micrograms of carbon in the same fashion into the relevant analysis chamber as the 20 ppm - 200 ppm methods.

µg Carbon

In this column the amount of carbon being analyzed will be automatically calculated for a given concentration and Method ID.

WARNING!

If µg Carbon value is higher than 100µg then the specific standard contains too much carbon for the NDIR to analyze. Please modify either the concentration or the Method ID for the standard.

Comments

Specific comments for the standard to be run may be entered here.

Saving the Calibration Table

To save a calibration table, click on **Save** in the **File Menu** or click **OK**. User will be asked if the calibration table should be saved.

OK Button

Will prompt user to save any unsaved changes before exiting screen.

Cancel Button

Will exit screen without saving any changes.

6.13 Calibration - Set Active

In the following TC, IC, TOC Curve choice, user will be selecting which calibration curve will be used for a given mode and range by clicking to the relevant pull down menu or display box. When finished, user must click the **OK** or **Cancel** button as appropriate.

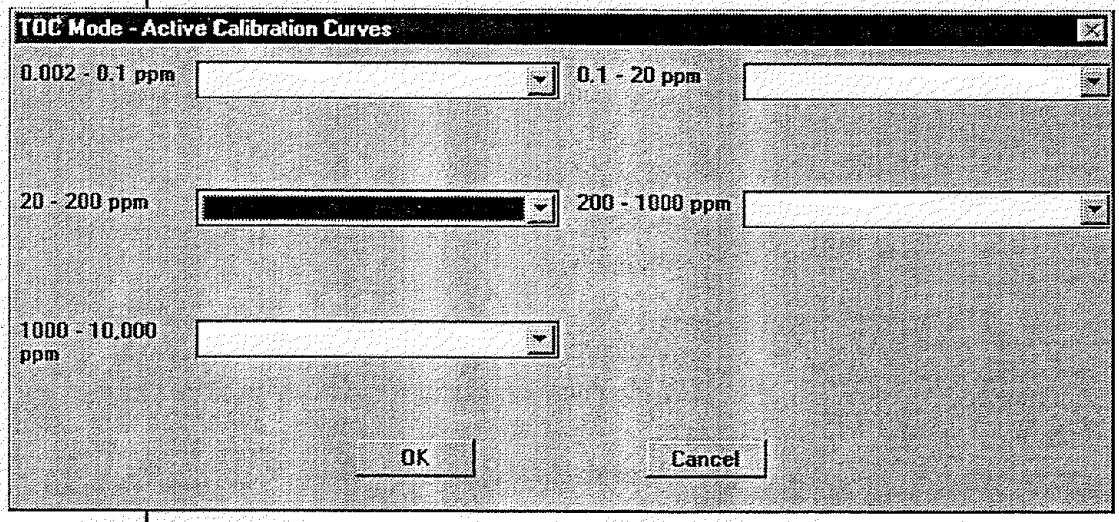


Figure 6-14 Calibration - Set Active Screen

NOTE: To run standards for a specific calibration curve, the user must select that curve as active in at least one range of one mode. Later in Run Setup, the user will go to that range and mode for standard selection.

6.14 Cal.Verification

In this screen the user will set up all possible calibration verifications needed for their application.

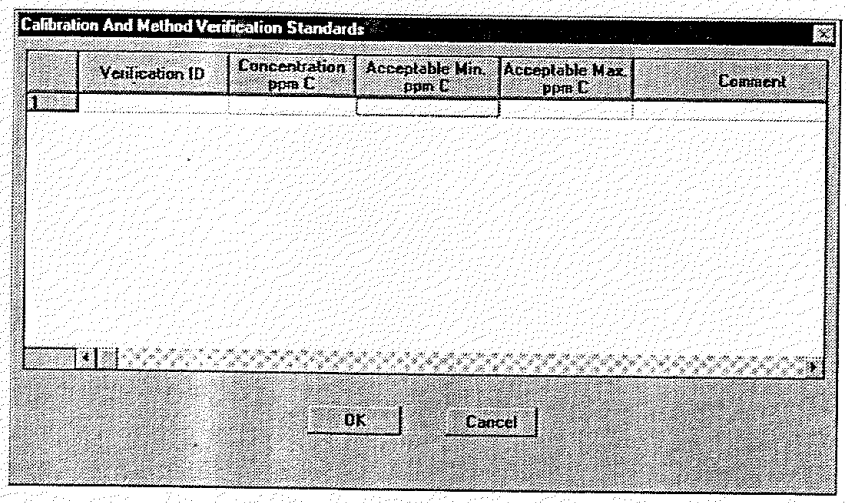


Figure 6-15 Calibration and Method Verification Screen

Shortcut Key

User can get more rows by pressing the down arrow on the PC keyboard.

NOTE: Running standards as calibration verification is more accurate than running standards as samples since this type of measurement accounts for the carbon in the preparation water used. (See 6-4 "Understanding Calibration")

Verification ID

Identification for the standard verification; may contain up to 32 characters.

Concentration ppm C

Concentration of the standard in parts per million carbon.

Acceptable Min. ppm C

Minimum acceptable concentration reported in analysis. If standard is reported below this value, "Out of Range" will be displayed in the results message box.

Acceptable Max. ppm C

Maximum acceptable concentration reported in analysis. If standard is reported above this value, "Out of Range" will be displayed in the results message box.

Comments

User defined comments for specific samples can be entered here.

6.15 About...

Displays general information about software including Version Number.

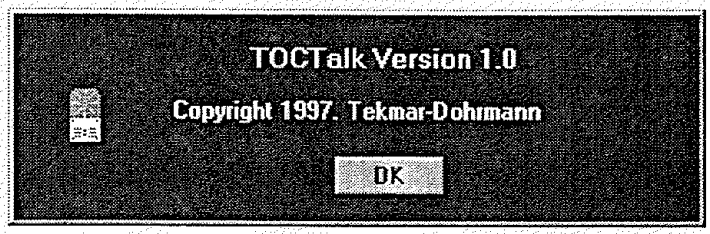


Figure 6-16 About TOC Talk Screen

6.16 Run Screen Choices

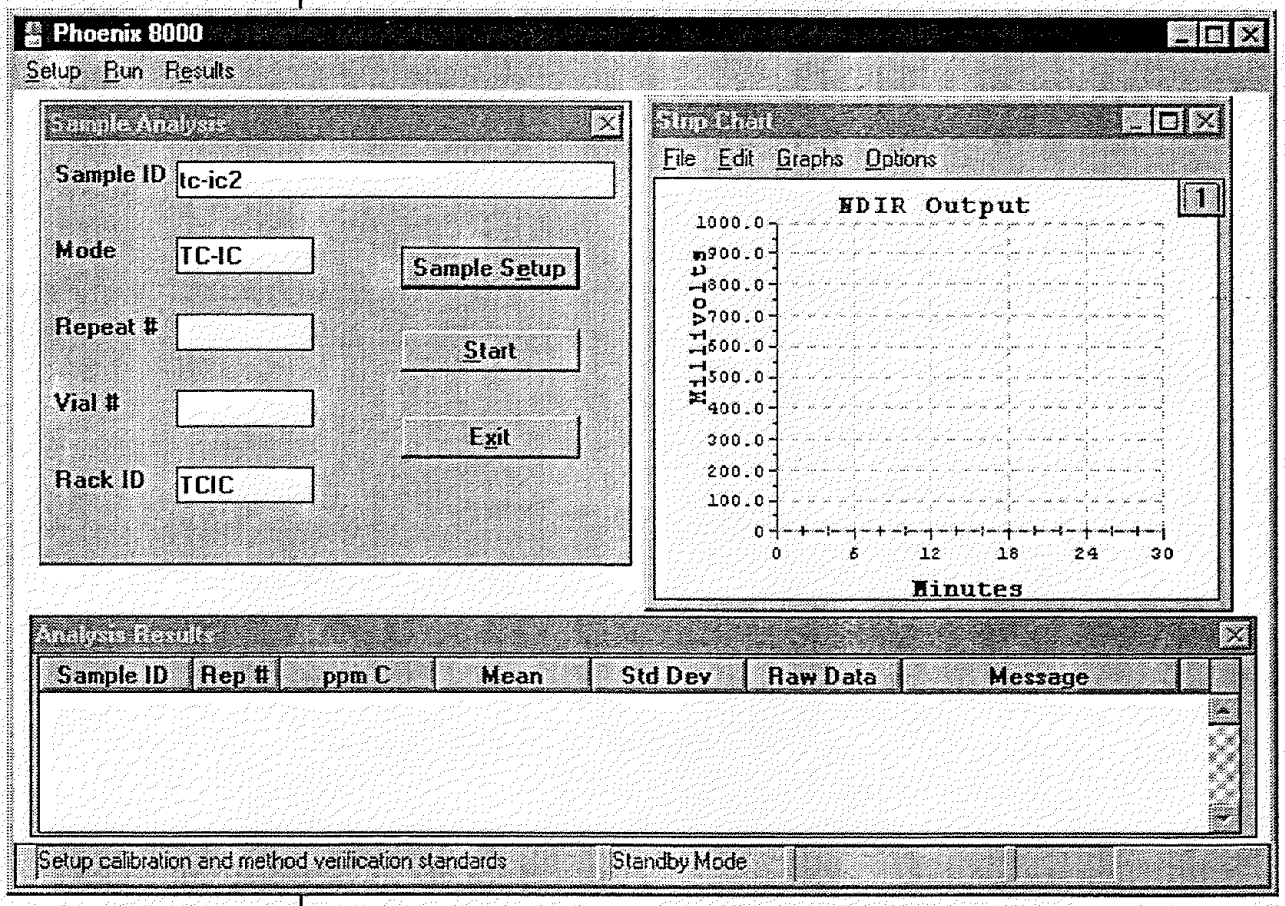


Figure 6-17 Run Screen

NOTE: Although you may increase screen resolution beyond 640x480, the Run screen may appear disoriented. If this occurs, simply move each of the three screens to the location you desire.

Run Window

The run window has three separate screens: Sample Analysis, Strip Chart, and Analysis Results.

Sample Analysis

This screen displays relevant information about either the current sample being run or the last sample finished if instrument is idle. Display boxes in this screen are read only:

Sample ID

Displays user defined identification of relevant sample.

Mode

Displays analysis mode of sample.

Repeat

Displays current replicate of sample being run or last sample run if instrument is idle.

Vial

Displays current vial Autosampler is analyzing or last vial analyzed if instrument is idle. This feature is grayed out in Automatic Syringe Mode.

Rack ID

Displays current rack configuration identification or last rack configuration run if instrument is idle. This feature is grayed out in Automatic Syringe Mode.

Sample Setup Button

Clicking this button will bring up the **Autosampler Setup** in **Autosampler Mode** or **Analysis Setup** in **Automatic Syringe Mode.**

Start/Stop Button

Once setup is complete, user will click the **Start** button to start the run. After clicking **Start,** button will become the **Stop** button. If, during a run, the user needs to stop the analysis for any reason click the **Stop** button. This will bring up a screen giving the user the following choices:

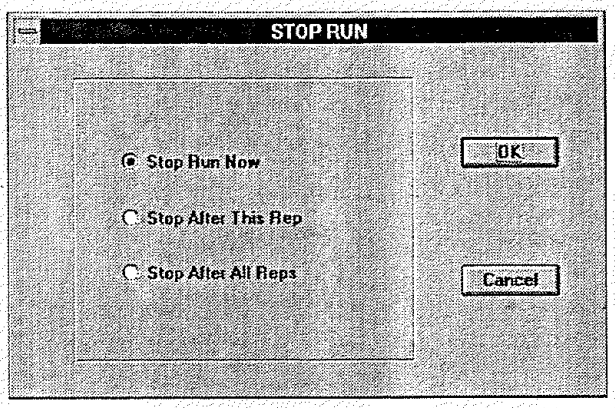


Figure 6-18 Start/Stop Options Screen

Stop Run Now

Stops run immediately. Instrument will automatically run a reset procedure to empty 25 ml from the UV Reactor and the IC Sparger.

Stop After This Rep

Stop Run After Current Analysis or Replicate. Instrument will automatically run a reset procedure to empty 25 ml from the UV Reactor and the IC Sparger.

Stop After All Reps

Stops Run After all replicates of current sample. Only relevant in Autosampler Mode. Instrument will automatically run a reset procedure to empty 25 ml from the UV Reactor and the IC Sparger.

OK Button

Exits the Run Window and closes all three screens.

Strip Chart Screen

The Strip Chart displays, in real time, response from the NDIR as Time verses Millivolts and is refreshed at the beginning of a sample run. Some analyses may require the Strip Chart to be re-scaled to be seen properly. To change the scale of the X and Y axis, double click on the appropriate axis. However, these modifications are only valid for the current screen. Once the Exit button is clicked, the strip chart reverts to default conditions.

Vertical Axis Screen

Displays voltage output from the NDIR in millivolts. The following is a description of important features on this screen:

- **From** Starting point on vertical axis in millivolts
- **To** Ending point on vertical axis in millivolts
- **Ticks-Step** Number of millivolts between vertical axis numeric headings

Horizontal Axis Screen

Displays Time in minutes. The following is a description of important features on this screen:

- **Range** Ending point of the time scale in minutes.
- **Ticks-Step** Number of minutes between numerical time headings

NOTE: There are other user defined features in the Strip Chart. Since they are not overly relevant to analysis, they are not described in this technical manual.

Analysis Results

This screen keeps a continuous display of analyses in process allowing the user to quickly and easily monitor instrument performance. this displays holds a maximum of 30 rows of data.

Sample ID

Identification of sample.

Rep

Replicate number of sample.

ppm C

Concentration in parts per million carbon of sample.

Mean

Displays the average of the replicates, where relevant, analyzed in parts per million carbon for samples and Calibration Verifications and in Area Counts for System Blanks and Standards.

Std Dev

Displays the standard deviation of the replicates, where relevant, analyzed in parts per million carbon for samples and Calibration Verifications and in Area Counts for System Blanks and Standards.

Raw Data

Area Counts from NDIR detector signal integration in mV~seconds.

Message

Relevant messages about the sample such as "Endpoint Timeout", "No Sample Detected", and "Out of Range".

Other Run Window Features

The bottom bar of the Run Window contains the additional features from left to right.

Event List Display

The bottom left corner will continuously display steps in the analysis currently being run and/or display the last non-run menu screen the user entered.

Mode Display

Displays current mode of the instrument (Running, Standby, and Ready).

NDIR Signal Display

Displays a continuously updated NDIR signal in millivolts.

NOTE: If this signal fails to update, the instrument has lost communication with the PC. This is an easy way to detect this failure.

WARNING!

No error message will be displayed in the event of a loss of autosampler communication with the Phoenix 8000. If you suspect such a failure, (i.e., running event display on run screen stops at an autosampler command) please check all STS 8000 power and RS-232 connections

Autosampler Setup

In this screen the user will program autosampler racks for analysis runs in Autosampler Mode when the **Sample Setup** button in clicked on from the **Run Window**. This screen *will not* appear in Automatic Syringe Mode.

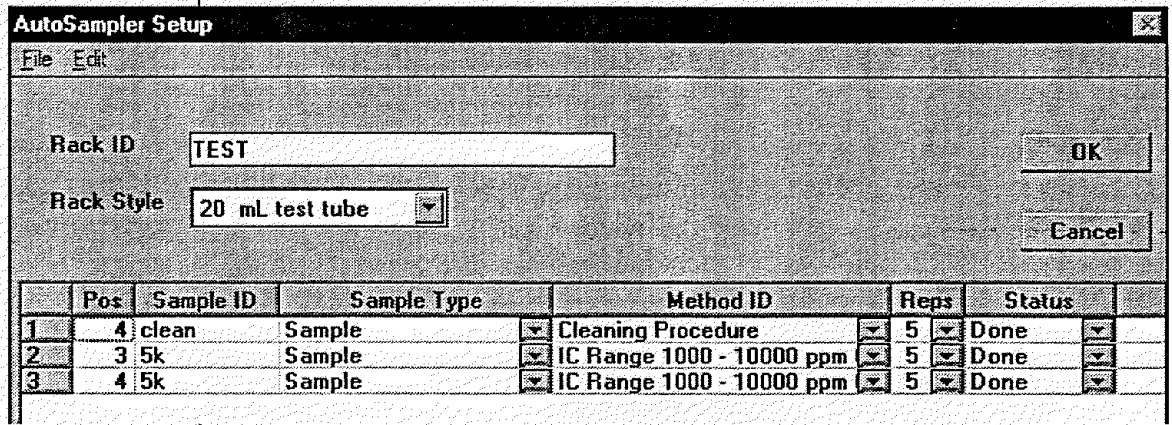


Figure 6-19 AutoSampler Setup

Shortcut Key

User can get more rows by pressing the down arrow on the PC keyboard. Rows generated in this fashion will copy the information of the row preceding it. The first number in a **Sample ID** will increase by an increment of 1 each time a row is added.

Examples: TOC1 becomes TOC2

10ppm becomes 11ppm

#1 10ppm becomes #2 10ppm

Shortcut Key

User can delete rows by simply pressing the Delete key.

Rack ID

Displays user defined configuration identification for a specific rack setup; may contain up to 8 characters.

Rack Style

Displays types of racks to be programmed. Click on pull down menu or display box to select a different Rack Style.

Pos

Position on the Autosampler where the sample vial is located.

Sample ID

Identification of the sample; may contain up to 32 characters.

Sample Type

One of the following sample types to be analyzed.

Sample

Use for sample analyses in any mode.

TOC, TC, IC Standard

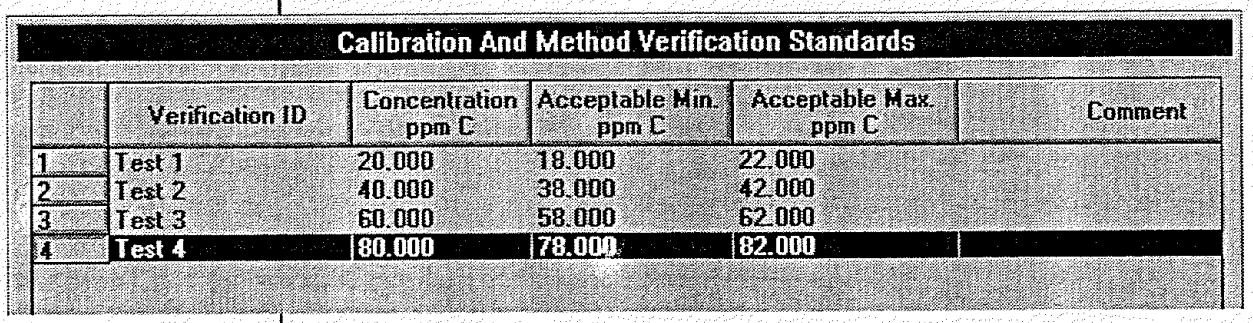
Instrument will prompt user for Range and specific standard to be run. Once a standard is chosen, the **Sample ID** and **Method ID** are automatically entered in the **Autosampler Setup.**

WARNING!

DO NOT change the Sample ID or Method ID cells for a specific standard after it has been chosen. These changes must be made in the Calibration Setup Screen. If you accidentally change these cells, choose the standard again to reset the cells to the correct configuration.

Cal. Verification

Instrument will prompt the user for the specific **Calibration Verification** required from the **Calibration Verification Setup Screen.**



Calibration And Method Verification Standards

	Verification ID	Concentration ppm C	Acceptable Min. ppm C	Acceptable Max. ppm C	Comment
1	Test 1	20.000	18.000	22.000	
2	Test 2	40.000	38.000	42.000	
3	Test 3	60.000	58.000	62.000	
4	Test 4	80.000	78.000	82.000	

Figure 6-20 Calibration Verification Screen

WARNING!

DO NOT change the Sample ID cell for a specific calibration verification after it has been chosen. These changes must be made in the Calibration Verification Setup Screen. If you accidentally change this cell, choose the Calibration Verification again to reset the cell to the correct configuration.

WARNING!

All blank sample types must be paired with their corresponding methods.

TC Blank Range 1

To analyze the blank for TOC, TC, TC in TC-IC, TC-IC in Range 2 ppb - 100 ppb.

TC Blank Range 2

To analyze the blank for TOC, TC, TC in TC-IC,TC-IC in Range 0.1 ppm - 20 ppm.

TC Blank Range 3,4,5

To analyze the blank for TOC, TC, and TC in TC-IC in Ranges 20 ppm - 10000ppm.

IC Blank Range 1

To analyze the blank for IC and TC in TC-IC in Range 2 ppb - 100 ppb.

IC Blank Range 2

To analyze the blank for IC, TC in TC-IC, and TC-IC in Range 0.1 ppm - 20 ppm.

IC Blank Range 3,4,5

To analyze the blank for IC, TC in TC-IC, and TC-IC in Range 20 ppm - 10000 ppm.

NOTE:

Default methods for range 3, 4, & 5 have the same blank type.

NOTE:

Blank Ranges 1 and 2 use DI Water to represent the sample volume in analysis. As a result, the first replicate for these ranges is inaccurate by the carbon contribution in the DI Water. Replicates after the first analysis, recycle the relatively "carbon free" DI Water from the previous replicate for an accurate measurement. Also, since system blanks in any range are often significantly less concentrated than previous runs of other sample types, Tekmar-Dohrmann recommends that the user perform 5 or more (maximum 10) replicates for blanks. The last 3 results are automatically stored for later use in Sample Analysis computation in the System Blanks Results Screen.

NOTE:

Keep in mind that Blank Methods do not need sample but do need an Autosampler position for the needle to move to. Tekmar-Dohrmann recommends using 1001.

Method ID

Displays method used for specific sample. Double click on this cell to view the **Method Setup Screen.**

WARNING!

Make sure if a blank method is chosen that the same blank sample type is chosen.

Cleaning Procedure

Match this choice with the "Sample" Sample Type to Rinse IC Sparger and UV Reactor with DI Water on the first replicate and carbon purged DI Water on further replicates. Tekmar-Dohrmann recommends 5 replicates for this procedure.

NOTE: Please remember that the Cleaning Method does not need sample but does need an Autosampler position for the needle to move to. Use position 1001.

Reps

Number of Replicates to be performed from a particular vial. Blanks require 5 or more reps for accurate results. When running samples, standards, or calibration verifications remember the amount of sample in the vial when choosing number or replicates.

Status

The Autosampler will only perform analyses on vials with a "Ready" status skipping all other status's. Other terms found in this box are:

- **Skip** User selectable, autosampler will not perform analyses on vial with Skip status.
- **Empty** User selectable, autosampler will not perform analyses on vial with Empty status.
- **Running** When viewing the Autosampler setup screen during an analysis one vial will display Running status.
- **Done** When vial is finished running it displays a Done status.
- **Canceled** When the analysis on a vial is stopped prematurely it will display a Canceled status.

Comments

Cell for user defined comments.

6.17 Analysis Setup

In this screen the user can setup the sample to run. This screen will not appear in Autosampler Mode. To access this screen, double-click the **Sample Setup** button in the **Run--Sample Analysis** window.

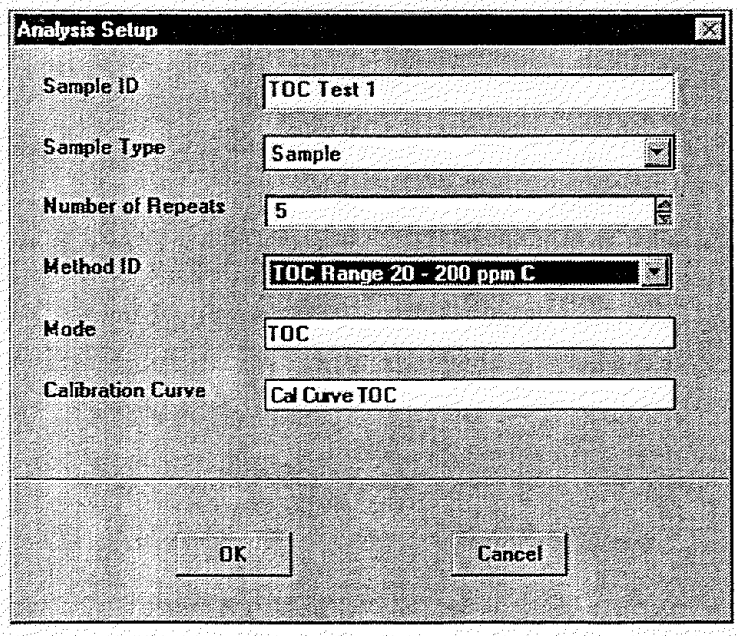


Figure 6-21 Analysis Setup Screen

Sample ID

Identification of the sample; may contain up to 32 characters.

Sample Type

One of the following sample types to be analyzed:

TOC, TC, IC Standard

Instrument will prompt user for **Range** and **specific standard** to be run. Once a standard is chosen, the **Sample ID** and **Method ID** are automatically entered in the **Autosampler Setup.**

WARNING!

DO NOT change the Sample ID or Method ID cells for a specific standard after it has been chosen. These changes must be made in the Calibration Setup Screen. If you accidentally change these cells, choose the standard again to reset the cells to the correct configuration.

Cal. Verification

Instrument will prompt the user for the specific **Calibration Verification** required from the **Calibration Verification Setup Screen.**

WARNING!

DO NOT change the Sample ID cell for a specific calibration verification after it has been chosen. These changes must be made in the Calibration Verification Setup Screen. If you accidentally change this cell, choose the Calibration Verification again to reset the cell to the correct configuration.

TC Blank Range 1

To analyze the blank for TOC, TC, TC-IC in Range 2 ppb - 100 ppb.

TC Blank Range 2

To analyze the blank for TOC, TC, TC-IC in Range 0.1 ppm - 20 ppm.

TC Blank Range 3,4,5

To analyze the blank for TOC, TC, TC-IC in Ranges 20 ppm - 10000ppm.

IC Blank Range 1

To analyze the blank for IC, TC-IC in Range 2 ppb - 100 ppb.

IC Blank Range 2

To analyze the blank for IC, TC-IC in Range 0.1 ppm - 20 ppm.

IC Blank Range 3,4,5

To analyze the blank for IC, TC-IC in Range 20 ppm - 10000 ppm.

NOTE:
Default methods for range 3, 4, & 5 have the same blank type.

NOTE:
Blank Ranges 1 and 2 use DI Water to represent the sample in analysis. As a result, the first replicate for these ranges is inaccurate by the carbon contribution in the DI Water. Replicates after the first, reuse the relatively "carbon free" DI Water from the 1 replicate for an accurate measurement. Also, since system blanks in any range are often significantly less concentrated than previous runs of other sample types, Tekmar-Dohrmann recommends that the user perform 5 or more replicates for blanks. The last 3 results are automatically stored for later computation in the System Blanks Results Screen.

Number of Repeats

Number of Replicates to be performed for an analysis. Blanks require 5 or more reps for accurate results. When running samples, standards, or calibration verifications, remember the amount of sample when choosing number of replicates.

Method ID

Displays method used for specific sample. Double click on this cell to view the **Method Setup Screen.**

NOTE: Make sure if a blank method is chosen that the same blank sample type is chosen.

6.18 Method Setup Description of Terms

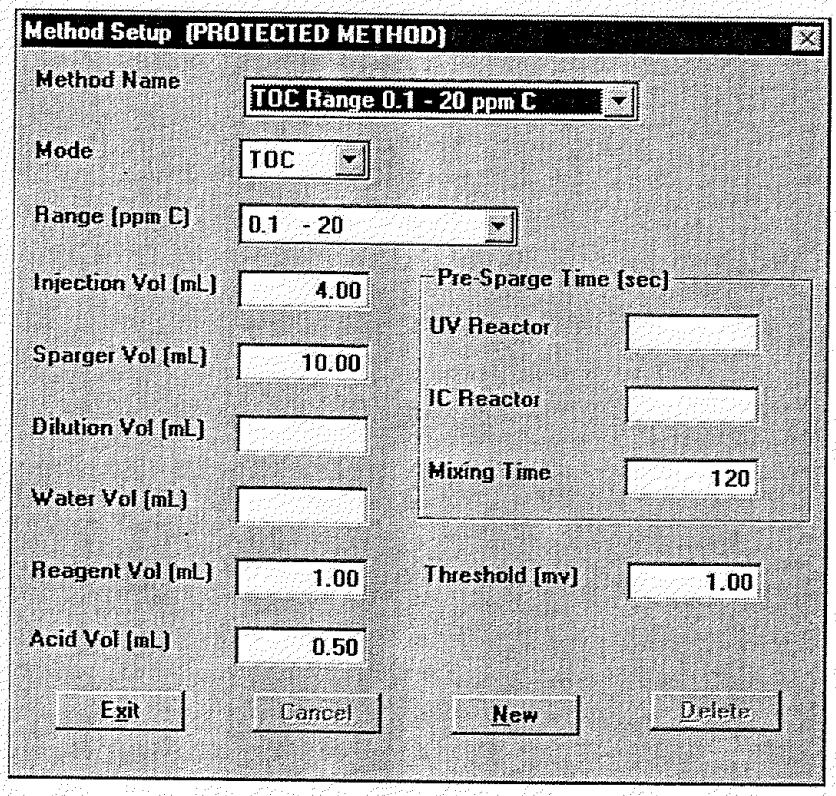


Figure 6-22 Method Setup Screen

WARNING!

Setting up New Methods should only be created by advanced users of the Phoenix 8000. Erroneous method setups may damage your unit.

Method Name

Identification for method setup when the user chooses Method ID in the Automatic Syringe or Autosampler Analysis Setup screens.

Mode

Type of Analysis being performed. Mode plus Range designates the Event List to be run.

NOTE: Every analysis needs an Event List and a Method Setup list of parameters to be executed. The Event List is a list of commands that the software uses to run an analysis. The instrument has only 5 Event Lists, based on concentration for every Analysis Mode. Where relevant, the Event List will reference the set of variables displayed in the Method Setup Screen. The user can define as many Method Setup sets of variables as necessary. This format allows for overall consistency in analysis while giving the user complete control over the critical chemistry aspects of the analysis.

Range (ppm C)

Concentration Range. The flexability of the syringe pump enables the user to overcome the limits of the NDIR by simply selecting different methods to cover different concentration ranges. Mode plus Range designates the Event List to be run.

NOTE: Custom methods with different method setup variables can affect the validity of the range displayed here.

Example:

If a custom method choosing a TOC Mode and a Range of 2ppb- 100ppb changes the Injection Volume from 20ml to 10ml, then the concentration range increases by a factor of 2. It is always important to recognize that the NDIR is not limited by purely carbon concentration but by µg of carbon analyzed. µg of carbon analyzed is dependent on the concentration of the sample times the Injection Volume.

Injection Volume (ml)

- TOC, TC - the amount of sample delivered to the UV Reactor
- IC - the amount of sample delivered to the IC Sparger
- TC-IC - the amount of sample delivered to UV Reactor during TC analysis and the amount of sample delivered to the IC Sparger during IC analysis

NOTE: The Cleaning Procedure does not use sample. However, to display a rough ppm C result of the Rinsing Water analyzed, the volume of the Rinsing Water used is entered here. Changing this value in a custom method WILL NOT change the amount of Rinsing Water used only the equation by which the ppm C is derived. Because this is a specific application, this method does not have the same modification flexibility as other sample analyses.

Increasing Injection Volume

Increasing Injection Volume increases the amount of µg of carbon to the detector and, in general, increases precision of the measurement. This may also be useful if you are trying to build a method with a range between 2 default methods. However, be careful not to exceed 100 µg carbon when making an analysis. Doing so risks the NDIR seeing CO_2 concentrations above its linear response range of 0 - 1000 mV. The equation for µg of carbon to the detector is:

*µg of Carbon to NDIR = Injection Volume (ml) * Carbon Concentration (ppmC)*

Other Factors:

All Modes

Do not increase Injection Volume above 20 ml. Syringe will not be able to accommodate higher volumes.

All Modes

Make sure you have enough sample to meet the increase of the injection volume.

TOC (0.1ppm-20ppm) Mode

Make sure to increment Sparger Volume by the increase in Injection Volume.

TOC (20ppm-200ppm, 200ppm-1000ppm, 1000ppm-10000ppm)

Can increase injection Volume from 0.5ml to 2ml without changing Sparger Volume if you run 3 or less replicates.

Decreasing Injection Volume

Decreasing Injection Volume will allow the use of less sample per analysis at the expense of a slightly less precise result. May be useful if trying to design a method whose range is in between 2 default methods. Tekmar-Dohrmann does not recommend Injection Volumes below 0.5 ml.

Other Factors:

TOC (2ppb-100ppb, 0.1ppm-20ppm)

To realize sample volume savings, you must change Sparger Volume by the decrease in Injection Volume.

TOC (20ppm-200ppm)

To realize sample volume savings, user must change Sparger Volume by the decrease in Injection Volume times the number of replicates.

Sparger Volume (ml)

The amount of sample delivered to the IC sparger for sample preparation.

TOC (2ppb-100ppb, 0.1ppm-20ppm)

Sparger Volume is the amount of sample needed in the IC Sparger to perform 1 replicate. The formula is the following:

Sparger Volume (ml) = Injection Volume (ml) + 4ml (For Rinsing)

TOC (20ppm-200ppm)

Sparger Volume is the sample required to perform up to 4 replicates.

TOC (200ppm-1000ppm, 1000ppm-10000ppm), TC (200ppm-1000ppm, 1000ppm-10000ppm), TC-IC (200ppm-1000ppm, 1000ppm-10000ppm)

Sparger Volume is the amount of sample diluted by the dilution volume to analyze highly concentrated samples.

TC (2ppb-100ppb, 0.1ppm-20ppm, 20ppm-200ppm), IC

Sparger Volume is not used.

Increasing Sparger Volume

By increasing Sparger Volume, you will be able to successfully increase Injection Volume for low concentration TOC analysis. Increasing Sparger Volume for high concentration TOC, TC analysis will allow you to change the ratio of your dilution.

Other Factors:

TOC (200ppm-1000ppm, 1000ppm-10000ppm), TC (200ppm-1000ppm, 1000ppm-10000ppm), TC-IC (200ppm-1000ppm, 1000ppm-10000ppm)

Make sure that your dilution allows no more than 100ug of carbon to be analyzed in the UV Reactor.

Decreasing Sparger Volume

Decreasing Sparger Volume will allow you to realize the sample savings of a decrease in Injection Volume for lower TOC concentrations. For higher TOC, TC concentrations, decreasing Sparger Volume will allow the user to change the dilution ratio.

Other Factor(s):

TOC (200ppm-1000ppm, 1000ppm-10000ppm), TC (200ppm-1000ppm, 1000ppm-10000ppm), TC-IC (200ppm-1000ppm, 1000ppm-10000ppm)

Dilutions will only be accurate if the amount of carbon in the dilution water is negligible compared to the sample carbon content. Make sure to only decrease the Sparger Volume in cases where it is needed to allow the amount of carbon in the UV Reactor to be less than 100µg.

Dilution Volume (ml)

TOC (200ppm-1000ppm, 1000ppm-10000ppm), TC (200ppm-1000ppm, 1000ppm-10000ppm), IC (200ppm-1000ppm, 1000ppm-10000ppm), TC-IC (200ppm-1000ppm, 1000ppm-10000ppm)

Dilution Volume is the amount of DI water used to dilute the sample.

NOTE: All other methods do not use Dilution Volume as a variable.

Increasing or Decreasing Dilution Volume

Changing the Dilution Volume changes the dilution ratio. Make sure that you do not increase the Dilution so much that the carbon content of the rinsing water becomes significant in analysis. Make sure not to decrease the Dilution Volume to the point that you exceed 100 µg carbon going to the NDIR.

$$\text{Dilution Factor} = \frac{\text{Sparger Volume}}{\text{Dilution Volume} + \text{Sparger Volume}}$$

Water Volume (ml)

The amount of DI water added to the reactor or sparger to provide a minimum level of reaction fluid for the analyses.

TOC (20ppm-200ppm, 200ppm-1000ppm, 1000ppm-10000ppm), TC (20ppm-200ppm, 200ppm-1000ppm, 1000ppm-10000ppm), IC (20ppm-200ppm, 200ppm-1000ppm, 1000ppm-10000ppm), TC-IC (20ppm-200ppm, 200ppm-1000ppm, 1000ppm-10000ppm)

In cases where the Injection Volume is less than 4ml, it may become necessary to add relatively carbon-free rinsing water to provide enough total volume in the relevant analysis chamber for an accurate carbon content reading. In the default methods that have a 0.5 ml sample injection volume, Tekmar-Dohrmann recommends the addition of 5 ml of rinsing water to allow the sample to reach the UV coils in the UV Reactor for TOC, TC analysis or to reach the fritted sparger in the IC Sparger for IC analysis.

TC, IC Blank Ranges 1 & 2

The amount of rinsing water needed to mimic the sample injection for these methods.

NOTE: At these low concentration ranges, it is important to analyze the system under conditions as close to the real analysis as possible. For this reason, use rinsing water to take the place of the sample in these analyses. Experienced users will recognize that this adds a source of carbon to the analysis that is not present in the actual sample run. To eliminate this factor from the system blank result the method recycles the ultra-clean water generated from the first replicate of the blank analysis for following replicates adding only new reagents. This is one reason why Tekmar-Dohrmann recommends a minimum of 5 (maximum of 10) replicates for this analysis since the software will only record the results of the last 3 replicates.

NOTE: All other modes do not use Water Volume.

Changing Water Volume

Change Water Volume to maintain at least a total of 5 ml of sample plus reagent plus rinsing water in the relevant analysis chamber.

Reagent Volume (ml)

TOC, TC, TC-IC

The amount of sodium persulfate used in the UV Reactor during analysis. The default setting is 1 ml.

NOTE: Not used in IC Analysis.

Changing Reagent Volume

Changing Reagent Volume changes the potency of the persulfate in breaking down carbon into CO_2. If you change the volume of persulfate, make sure to also change the amount of persulfate in the relevant TC blanking method and rerun your blanks. Otherwise, the carbon content of the reagent will be incorrectly accounted for.

Acid Volume (ml)

TOC, IC, TC-IC

The amount of acid used in the IC Chamber to break down Inorganic Carbon. The default setting is 0.5 ml.

NOTE: Not used in TC Analysis.

Changing Acid Volume

TOC

DO NOT significantly increase the amount of acid in this mode. Doing so can dilute your sample causing artificially lower results

IC, TC-IC

Changing Acid Volume changes the potency of the acid in breaking down carbon into CO_2. If you change the volume of acid, make sure to also change the amount of acid in the relevant IC blanking method and rerun your blanks. Otherwise, the carbon content of the acid will be incorrectly accounted for.

UV Reactor Pre-Sparge Time (sec)

The delay time after the reagent is delivered to the UV reactor.

TOC, TC, TC-IC

Sometimes the UV Lamp is so powerful in breaking down carbon into CO_2 that the NDIR will see a significant reaction before the persulfate even enters the UV Reactor if the carrier gas remains on. The result can be a "double peak". Though the area of this peak is rigorously correct, it is preferable to send all of the CO_2 generated to the NDIR at the same time with a single peak result. To accomplish this, the instrument stops carrier gas flow briefly after the beginning baseline is measured.

NOTE: Not used in IC Analysis.

Changing UV Reactor Pre-Sparge Time

DO NOT significantly lengthen the UV Reactor Pre-Sparge Time. Long periods of stopped flow can cause the NDIR baseline to fluctuate. If the particular type of sample being run does not need the default 10 seconds wait time, Tekmar-Dohrmann recommends that you decrease this value.

NOTE: TOC (2ppb-100ppb, 0.1ppm-20ppm) has a UV Pre-Sparge of 0 seconds. In these two ranges, emptying the IC Sparger after the introduction of persulfate, but before starting the carrier gas, compensates for the UV-Reactor pre-sparge time.

IC Pre-Sparge Time (sec)

The delay time after the reagent is delivered to the UV reactor.

IC, TC-IC

Sometimes there is a significant quantity of CO_2 dissolved in solution that would be liberated before the acid even enters the IC Sparger if the carrier gas remains on. The result can be a "double peak". Though the area of this peak is rigorously correct, it is preferable to send all of the CO_2 generated to the NDIR at the same time with a single peak result. To accomplish this, the instrument stops carrier gas flow briefly after the beginning baseline is measured.

NOTE: Not used in TOC, TC analysis.

Changing IC Pre-Sparge Time

DO NOT significantly lengthen the IC Sparger Pre-Sparge Time. Long periods of stopped flow can cause the NDIR baseline to fluctuate. If the particular type of sample being run does not need the default 10 seconds wait time, Tekmar-Dohrmann recommends you decrease this value.

Mixing Time (sec)

TOC

Mixing time represents the sparging time needed to remove all the Inorganic Carbon from the sample in the IC Sparger.

TC (200ppm-1000ppm, 1000ppm-10000ppm), IC (200ppm-1000ppm, 1000ppm-10000ppm)

Represents the time needed to mix the sample with the dilution water in these modes. Not needed in TOC analysis since sample is already mixing with dilution water during IC removal.

NOTE: Not used in other analyses.

Changing Mixing Time

TOC

The time requirement for IC removal is dependent on IC present in samples being run. The default setting is 120 seconds. The user can change this value as deemed appropriate.

Threshold (mV)

The measure of how close the NDIR signal has to be to the beginning baseline to end the analysis.

Example: If the beginning baseline is 2 mv and the threshold is 1 mv, then the analysis will end when the NDIR signal goes below 3 mV.

This value is also used in the software to determine if sample is present in the relevant analysis chamber. A "Sample Not Detected" message will be displayed if the peak height of the reaction does not exceed twice the threshold value within two minutes.

Setting the Threshold

Tekmar-Dohrmann recommends setting the threshold to roughly 1% of the user average peak height result. Higher threshold settings will cut analysis times. However, Tekmar-Dohrmann does not recommend setting the threshold at more than 5% of peak height. For the most accurate results, make sure the threshold setting is the same for your calibration and your sample runs.

6.19 Results

Single Analysis

The Single Analysis screen displays the results for Automatic Syringe Mode. This screen can be viewed from Multiple Analysis Screen for Autosampler Mode.

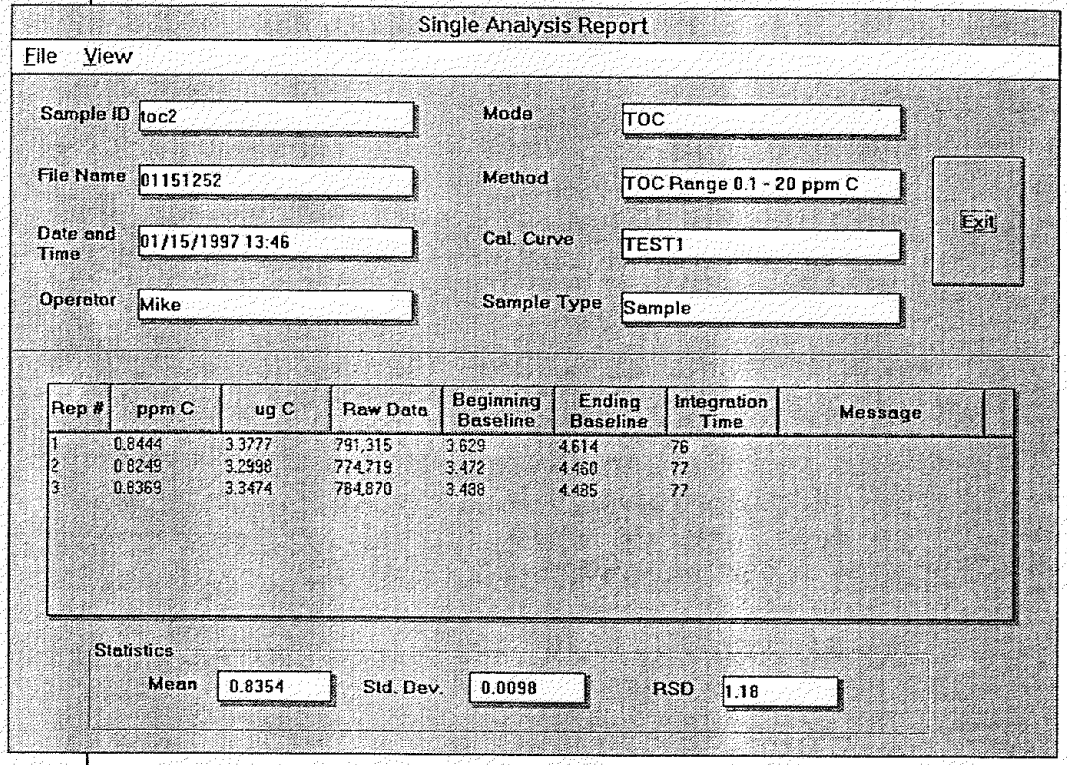


Figure 6-23 Single Analysis Report Screen

WARNING!

DO NOT try to view autosampler runs in this screen. Software will try to force the data from several vials into a one vial format. Please use the Multiple Analysis Screen to view autosampler results.

Sample ID

Identification of the sample.

WARNING!

User must enter identification here. Otherwise, all columns will shift to the left in the Multiple and Single Analysis Results screens.

File Name

File name the analysis is stored under. This is roughly based on the time of analysis.

Date and Time

Full date and time the analysis was performed.

Operator

Operator who performed the analysis.

Mode

Displays Analysis Mode

Method

Method ID of the analysis.

Cal. Curve

Calibration curve used in the analysis.

Sample Type

Type of sample being analyzed.

Rep #

The specific replicate of sample run.

ppm C

Part per million carbon concentration of the sample.

µg C

Micrograms of carbon in the sample.

Raw Data

Area Counts of sample analysis.

Beginning Baseline

Baseline reading, in millivolts, at the beginning of the sample analysis.

Ending Baseline

Baseline reading, in millivolts, at the end of the sample analysis.

NOTE: The difference between the beginning and ending baseline reading should approximately be the threshold value, in millivolts, set for the method run. If the baseline does not come back within this value, then the system reached its endpoint timeout.

Integration Time

Time, in seconds, for the actual sample analysis.

NOTE: Endpoint Timeout - If the analysis does not return to the beginning baseline within the threshold value after the Max. Integration Time then the instrument will report an Endpoint Timeout Message. In most cases, the result of the analysis is still accurate. In these cases, the user should view the data and the sample curve generated to decide on the validity of the result.

Mean

Displays the average of the replicates, where relevant, analyzed in parts per million carbon for samples and Calibration Verifications and in Area Counts for System Blanks and Standards.

Std. Dev.

Displays the standard deviation of the replicates, where relevant, analyzed in parts per million carbon for samples and Calibration Verifications and in Area Counts for System Blanks and Standards.

RSD

Displays the relative standard deviation, in percent, for the analysis. Defined as: *RSD = Std. Dev. / Mean * 100*

Viewing An Analysis Curve

To view the actual strip chart recording of a specific analysis, double-click on the relevant row of that analysis. The Strip Chart will automatically scale the X and Y axis.

Printing on the Single Analysis Screen

To send the data from the Single Analysis Screen to a printer, choose the print option in the **File** pull down menu.

Multiple Analysis

Choose this screen to view autosampler analysis runs or to prepare reports of different runs in Automatic Syringe or Autosampler Mode.

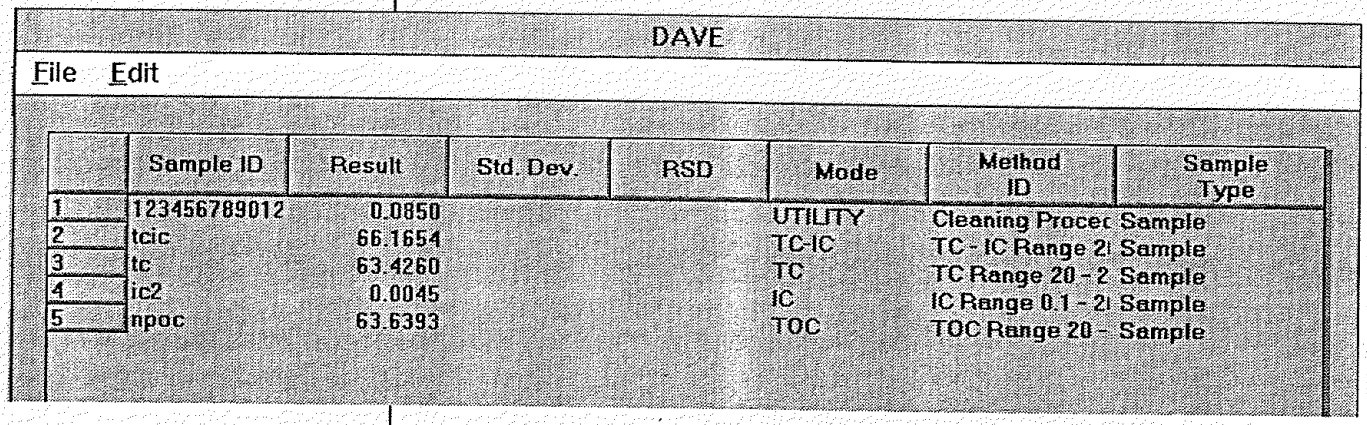


Figure 6-24 Multiple Analysis Screen

Sample ID

Identification of the sample.

Result

Displays the average of the replicates analyzed in parts per million carbon for samples and Calibration Verifications and in Area Counts for System Blanks and Standards.

Std. Dev.

Displays the standard deviation of the replicates, where relevant, analyzed in parts per million carbon for samples and Calibration Verifications and in Area Counts for System Blanks and Standards.

RSD

Displays the relative standard deviation, in percent, for the analysis. Defined as: *RSD = Std. Dev. / Mean * 100*

Mode

Displays Analysis Mode

Method ID

Method ID of the analysis.

Sample Type

Type of sample analyzed.

NOTE: To get the following columns use the scroll bar at the bottom of the screen.

Vial

Displays the position on the autosampler the sample vial was located.

Date and Time

Full date and time the analysis was performed.

Message

Displays any messages such as "Endpoint Timeout", "Out of Range", and "No Sample Detected" if relevant.

Reaching the Single Analysis Screen

Sometimes the user will need to view the Single Analysis data for a particular vial. Double-click on the specific vial's row to pull up a Single Analysis Screen of that sample.

Printing from the Multiple Analysis Screen

The user has two printing options in the **File** pull down menu for the Multiple Analysis Screen:

Print Summary Report

To print only the information on the Multiple Analysis Report select this option.

Print Detailed Report

To print the set of Single Analysis reports for the samples listed select this option.

Calibration

There is a two-part Calibration Results Screen that displays the data from the standards analyzed and the graph of the current calibration curve.

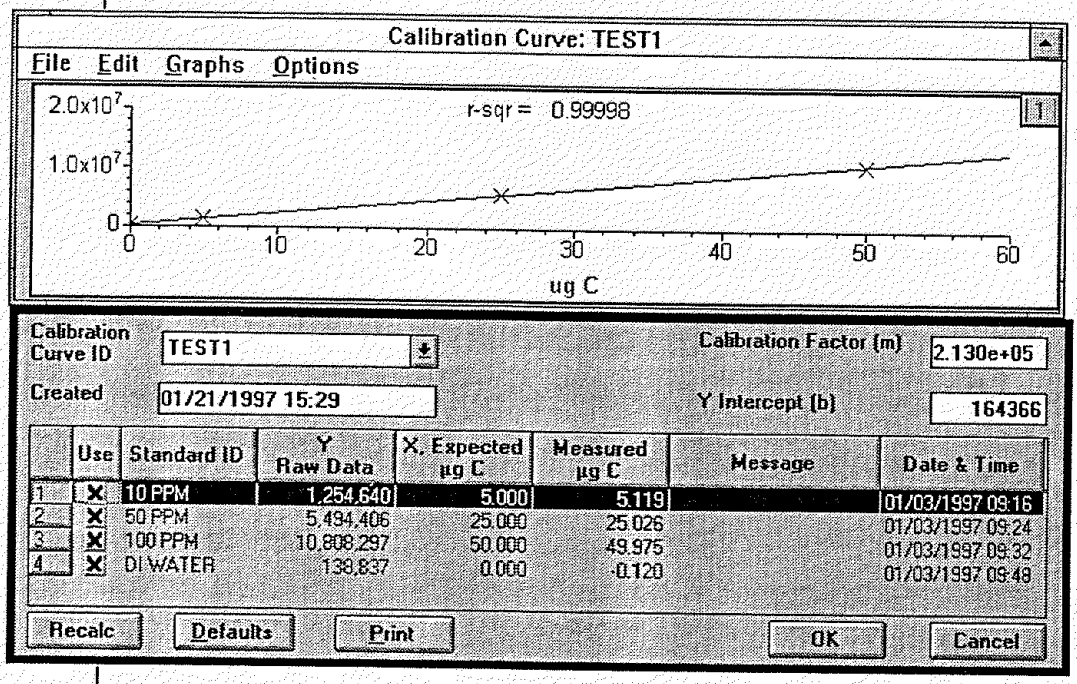


Figure 6-25 Calibration Curve Screen

Calibration Curve Screen

Displays, in a graph, the relationship between micrograms of carbon on the X axis and NDIR response in Area Counts on the Y axis. The calibration curve is the best linear fit of the standard data point selected. The r^2 factor for the curve is displayed toward the top center of the graph. Tekmar-Dohrmann recommends a r^2 value of 0.999 or greater depending on analysis requirements.

Printing Calibration Curve Screen

To print this graph, choose the printing option from the File pull down menu.

Calibration Setup Screen

Standard results can be reviewed and used to derive calibration curves for the instrument.

Calibration Curve ID

Identification for the calibration curve generated.

Created

Full date and time calibration curve was generated.

NOTE: This is NOT when the standards for the calibration curve were run, only when the standard data was used to make the calibration curve. The dates for standard runs are shown for each standard separately in the column furthest right on this screen.

Calibration Factor (m)

The response factor of micrograms of carbon versus Area Counts of NDIR output.

Y - Intercept (b)

The y value when "zero" measured carbon is analyzed. Represents constant sources of carbon in all reactions such as standard preparation water and reagents. See the description of calibration for more details.

Use

Place where the user can choose whether or not to include a particular standard in a calibration curve.

Standard ID

Identification of the standard.

Y Raw Data

Area Counts generated from the NDIR for the standard.

X, Expected mg Carbon

Expected amount of carbon expected in the standard.

Measured µg Carbon

For a given calibration curve, the amount of carbon measured for the standard.

Message

Displays any messages such as "Endpoint Timeout", "Out of Range", and "No Sample Detected" if relevant.

Date and Time

Full date and time the standard was analyzed.

Recalc button

Clicking this button recalculates the calibration curve for the standards currently selected.

Defaults button

Clicking this button resets the Response Factor (m) and the Y - Intercept to their default values.

Print button

Clicking this button prints the Calibration Setup Screen ONLY.

OK button

Clicking this button accepts the current calibration as correct.

Cancel button

Clicking this button exits the screen without saving the current calibration curve.

System Blanks Review Screen

In this screen the user can view system blank data for the instrument.

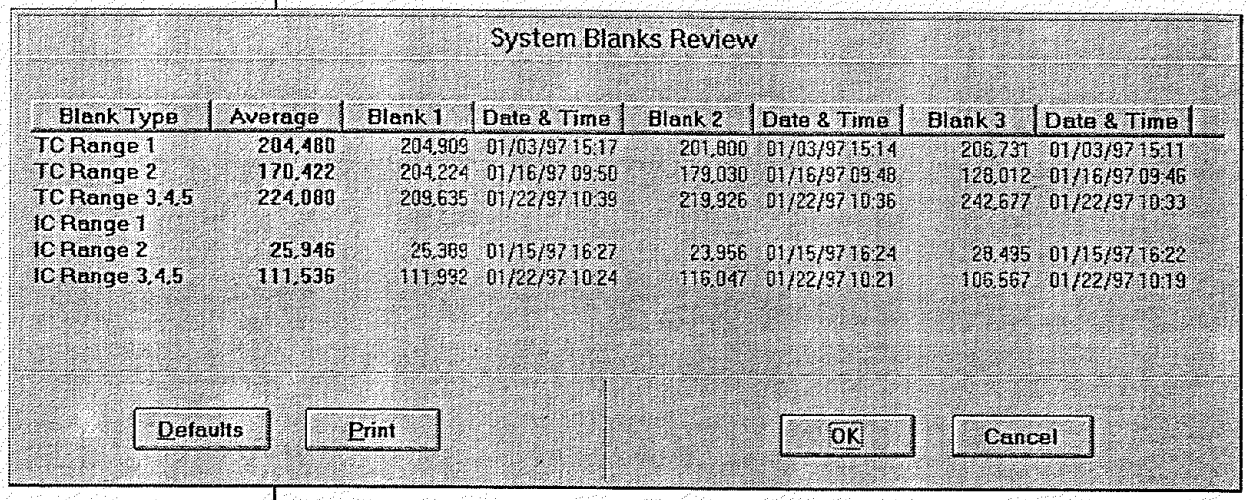


Figure 6-26 System Blank Review Screen

Data collection for blanks

The data from the last three blank analyses in any range will be displayed on this screen. The average of this data is used in the relevant sample analyses. This average is also displayed on this screen.

Defaults

Clicking this button clears the highlighted row on the system blanks table.

Print

Clicking this button prints the System Blanks Review Screen.

OK

Clicking this button accepts the data displayed on the screen.

Cancel

Clicking this button does not save the data displayed on the screen.

6.20 Opening TOC Talk Files Using Microsoft Excel

TOC Talk creates .prn files of your runs. These files are designed to work hand in hand with Microsoft Excel 5.x and higher. To open a .prn file:

1. **Start** the **Excel** program.
2. From the **File** menu, select **Open.**
3. In **Files of type, select [*.prn; *.txt; *.csv]**
4. Highlight the **.prn file** you wish to open.
5. Click **Open.**

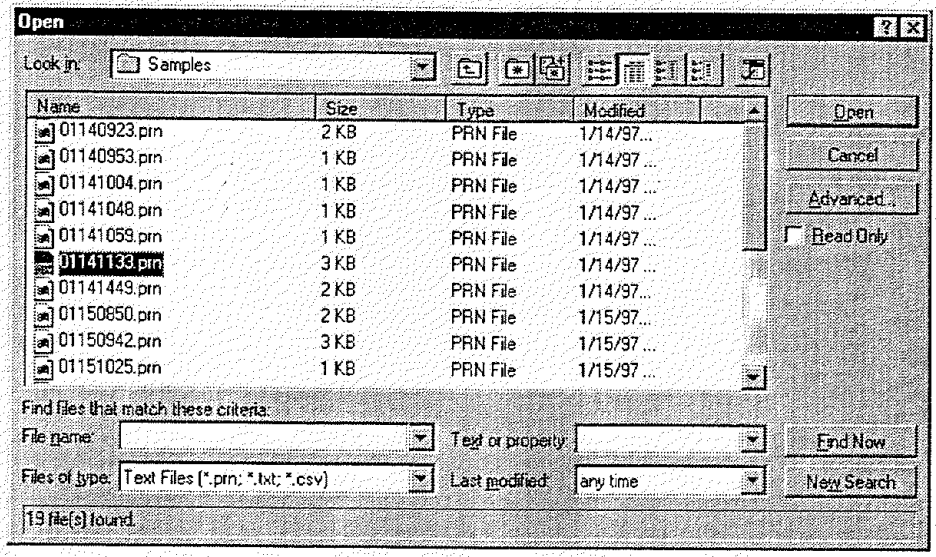


Figure 6-27 Open Excel .prn File

6. The **Text Import Window** will appear:

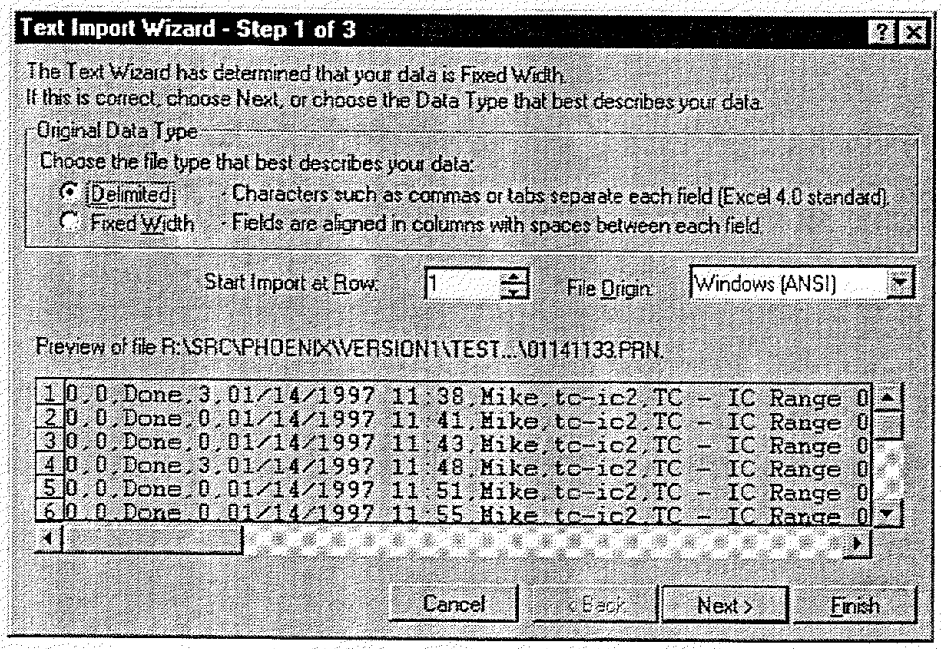


Figure 6-28 Excel Text Import Wizard - Step 1 of 3

7. Select the **Delimited** radio button **in Original Data Type.**
8. Make sure that **Windows (ANSI)** is selected as the **File Origin.**
9. Click **Next.** The next step will appear:

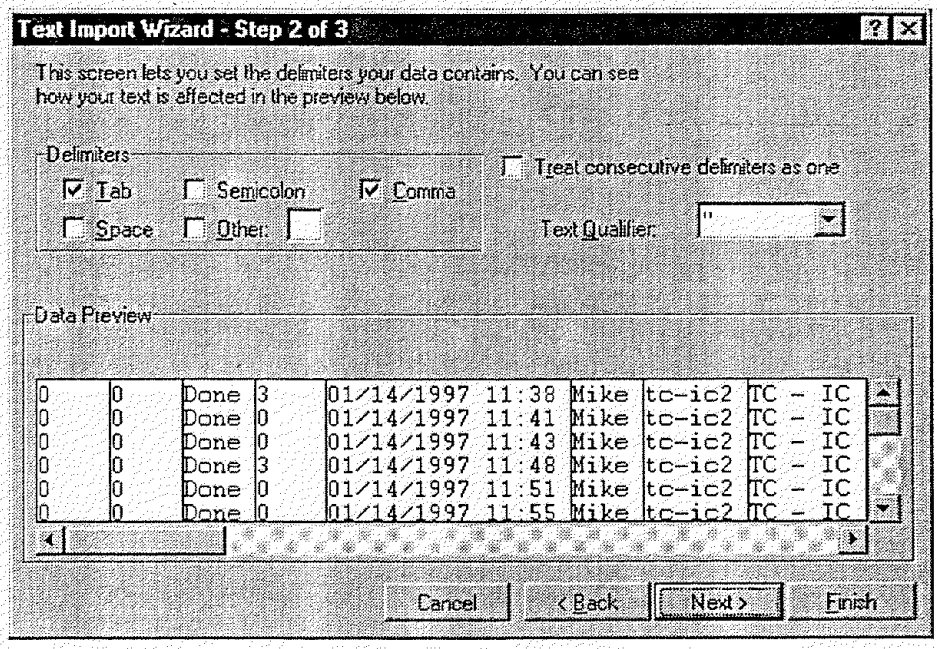


Figure 6-29 Excel Text Import Wizard - Step 2 of 3

10. In **Delimiters**, only the **Comma** option should be checked.
11. Click **Next.** The next step will appear:

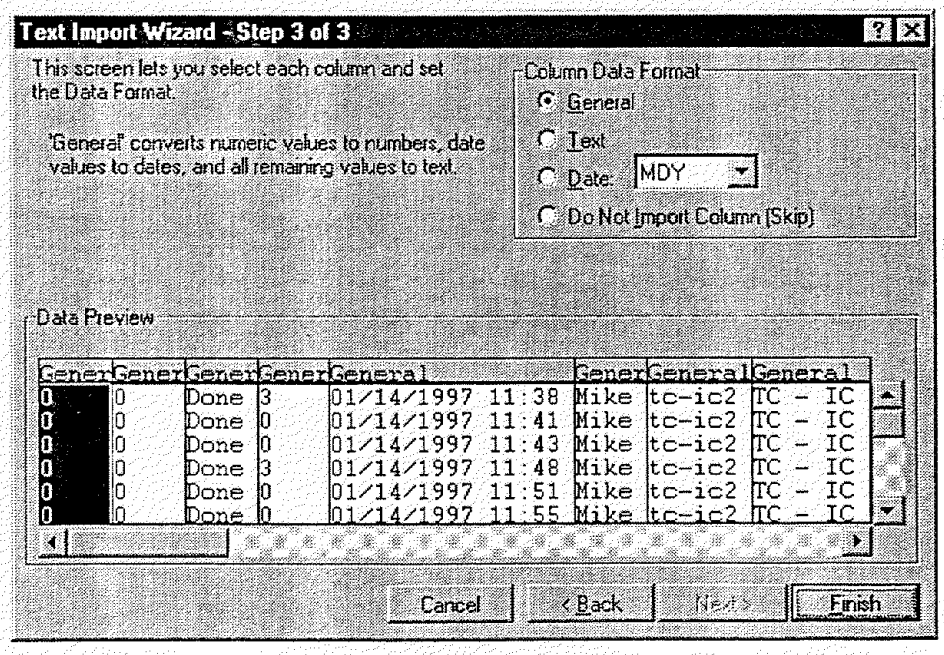
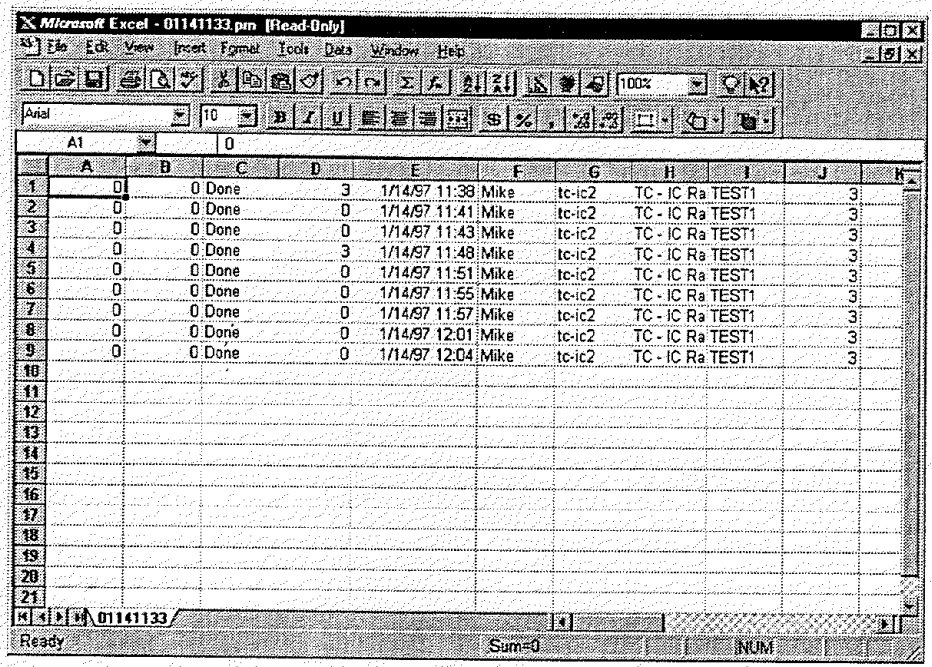


Figure 6-30 Excel Text Import Wizard - Step 3 of 3

12. Select the **General** radio button in **Column Data Format.**
13. Click **Finish.**
14. Your Excel .prn file will now appear. You may resize the fields to fit your individual specifications.



	A	B	C	D	E	F	G	H	I	J
1	0	0	Done	3	1/14/97 11:38	Mike	tc-ic2	TC - IC Ra	TEST1	3
2	0	0	Done	0	1/14/97 11:41	Mike	tc-ic2	TC - IC Ra	TEST1	3
3	0	0	Done	0	1/14/97 11:43	Mike	tc-ic2	TC - IC Ra	TEST1	3
4	0	0	Done	3	1/14/97 11:48	Mike	tc-ic2	TC - IC Ra	TEST1	3
5	0	0	Done	0	1/14/97 11:51	Mike	tc-ic2	TC - IC Ra	TEST1	3
6	0	0	Done	0	1/14/97 11:55	Mike	tc-ic2	TC - IC Ra	TEST1	3
7	0	0	Done	0	1/14/97 11:57	Mike	tc-ic2	TC - IC Ra	TEST1	3
8	0	0	Done	0	1/14/97 12:01	Mike	tc-ic2	TC - IC Ra	TEST1	3
9	0	0	Done	0	1/14/97 12:04	Mike	tc-ic2	TC - IC Ra	TEST1	3

Figure 6-31 Opened Excel .prn File

7.1 Overview

This chapter describes Phoenix 8000 maintenance and troubleshooting procedures.

7.2 Daily Maintenance Checks

The following components should be checked on a daily basis:

- Carrier gas, 500+ psi from tank to run scheduled load*
- Ample persulfate supply for load*
- Ample acid supply for load*
- Replaced and ample DI water supply for load*
- Check chlorine to ensure ample life for load*
- Carrier gas flow rate (200 cc/min ± 10%)
- Gas/Liquid Separator water level filled to waste outlet
- Empty Mist Trap
- Make sure 8-port valve thumbscrews are hand-tightened

*Load = estimated amount of daily analysis

7.3 Weekly Maintenance Checks

The following components should be maintenanced on a weekly basis:

- Daily checks, plus:
- Clean UV Reactor and IC Sparger with soap and water if needed
- Change reagents if needed

7.4 Monthly Maintenance Checks

The following components should be maintenanced on a monthly basis:

- Daily and weekly checks, plus:
- Change Chlorine Scrubber (p/n 14-7014-024)
- Inspect Permeation Dryer for damage, water accumulation (p/n 090-825)

NOTE: The Consumables Kit includes most items needed for preventative maintenance.

D, W, M	ACTION	DATE	DATE	DATE	DATE	DATE	DATE	DATE	DATE	DATE	DATE
Daily	Carrier Gas, 500+ psi from tank to run scheduled load										
	Ample persulfate supply for load										
	Ample acid supply for load										
	Replaced and ample DI Water supply for load										
	Check chlorine scrubber to ensure ample life for load										
	Carrier Gas Flow Rate (200cc/min ±10%)										
	Gas/Liquid Separator water level filled to waste outlet										
	Empty Mist Trap										
	Make sure 8-port valve thumbscrews are hand-tightened										
Weekly	Clean UV Reactor and IC Sparger with soap and water as needed										
	Change Reagents, if needed										
Monthly	Change Chlorine Scrubber										
	Inspect Permeation Dryer for damage, water accumulation										

Figure 7-1 Preventative Maintenance Check List

7.5 ExteriorLeak Checking of the Phoenix 8000

A leak check from the outside of Phoenix 8000 is performed by measuring the flow through three parts of the unit—the IC sparger, the UV reactor, and the moisture control system.

Leak Checking the IC Sparger

1. Verify that the gas supply to the IC sparger is on. From **Setup,** choose **Diagnostics** and **Valves.** Make certain that **Gas to IC Sparger** is turned on and **Gas to UV Reactor** is turned off.

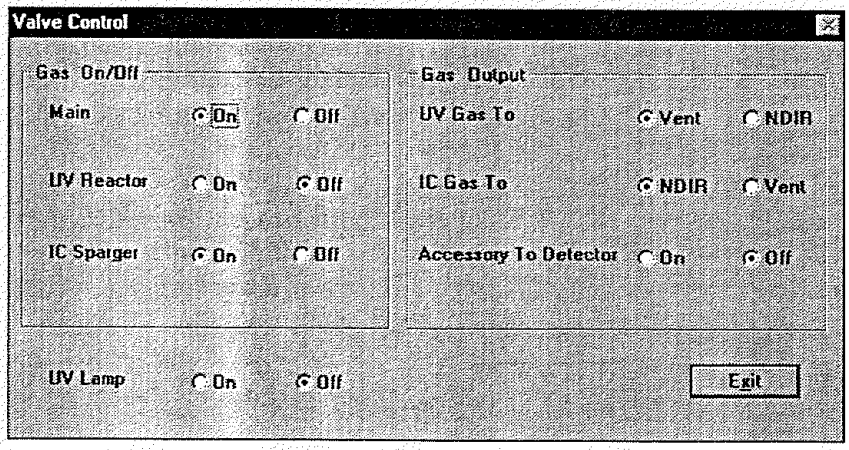


Figure 7-2 Diagnostic Screen

2. Locate the IC sparger. Check all rubber seals to verify they are intact and securely in place. Remove the Teflon tube with a red line entering the top and measure the flow through this tube with a hand-held flowmeter. If you obtain a reading of ±10%, continue to step 3.

   If you obtain a reading of less than 180 cc/min, see section 7.6, "Interior Leak Checking of the Phoenix 8000." It is likely that the IC sparger flow restrictor, IC gas valve, or main valve is leaking. Additionally, if the flow rate is off by more than 25%, you may need to perform a leak check.

3. In the **Diagnostics** screen, set the **IC Gas** to **NDIR** and set the **UV Gas** to vent.

4. Locate the gas/liquid separator. Check all rubber seals to verify they are intact and securely in place. Remove the green inlet tube at the top of the unit and measure the flow with a hand-held flowmeter. Again, you should obtain the same reading as in step 2.

   If you obtain a reading of less than 98% of step 2, remove the glass frit in the IC sparger. Soak the frit in basic solution of 1M NaOH and reinsert in sparger.

   Remeasure the flow entering the gas/liquid separator. If cleaning the IC sparger frit did not correct the problem, see section 7.6, "Interior Leak Checking of the Phoenix 8000."

   If you obtain a reading within 98% of step 2 entering the gas/liquid separator, continue to "Leak Checking the UV Reactor."

Leak Checking the UV Reactor

1. Verify that the gas supply to the UV reactor is on. From **Setup,** choose **Diagnostics** and **Valves.** Make certain that **Gas to UV Reactor** is turned on and **Gas to IC Reactor** is turned off.
2. Locate the UV reactor. Check all rubber seals to verify they are intact and securely in place. Remove the blue tube entering the top and measure the flow through this tube with a hand-held flowmeter. If you obtain a reading of 200 cc/min, continue to step 3.

   If you obtain a reading of 180-200 cc/min, adjust your teank pressure to achieve 200 cc/min. If you do not achieve 200 cc/min, see section 7.6, "Interior Leak Checking of the Phoenix 8000." It is likely that the UV reactor flow restrictor, gas valve, or main valve is leaking.

NOTE: Do not set pressure regulator above 35 psi. If your flow rate deviates 5 cc/min from the reading obtained in step 2, see section 7.6, "Interior Leak Checking of the Phoenix 8000".

3. In the **Diagnostics** screen, set the IC gas to vent and set the UV gas to NDIR.
4. Locate the gas/liquid separator. Check all rubber seals to verify they are intact and securely in place. Remove the green inlet tube at the top of the unit and measure the flow with a hand-held flowmeter. If you obtain a reading of 200 cc/min, continue to "Leak Checking the Moisture Control System."

   If you obtain a reading of less than 190 cc/min, see section 7.6, "Leak Checking Inside Phoenix 8000."

Leak Checking the Moisture Control System

1. Locate the mist trap. Check all rubber seals to verify they are intact and securely in place. Remove the top green tube and measure the flow. If you obtain a reading of 200 cc/min, continue to step 2.

   If you obtain a reading of less than 200 cc/min, check the DI water level in the gas/liquid separator. The water level should be even with the sidearm. Also check rubber seal.
2. Locate the chlorine scrubber. Check all rubber seals to verify they are intact. Remove the green tube entering the side of the scrubber with copper granules and measure the flow.

continued

If you obtain a reading of less than 200 cc/min, check the rubber seal on the mist trap outlet and the plug in the bottom of the mist trap. If you still obtain a reading of less than 200 cc/min, the problem may be a leak in the permeation tube connections. See section 7.6, "Interior Leak Checking of the Phoenix 800. "

If you obtain a reading of more than 200 cc/min at the scrubber inlet, the problem is definitely a break inside the permeation tube, which should be replaced.

7.6 Interior Leak Checking of the Phoenix 8000

If leak checking outside the unit presents a flow problem that can not be resolved outside Phoenix 8000, you must perform a leak check inside the unit. Leak checking inside Phoenix 8000 consists of measuring flows through a series of valves, tees, flow restrictors, and flowmeters located in the right bay of the unit. These components may be exposed by removing the right bay cover.

NOTE: The left bay contains electronic components and should not be removed unless instructed by Tekmar-Dohrmann to do so.

To remove the right bay cover, first move reagents, DI water, and waste containers away from the unit. Remove the two screws on the top of the cover and lift the panel off the unit.

Refer to Figure 7-3 for the location of valves, tees, flow restrictors, and flowmeters.

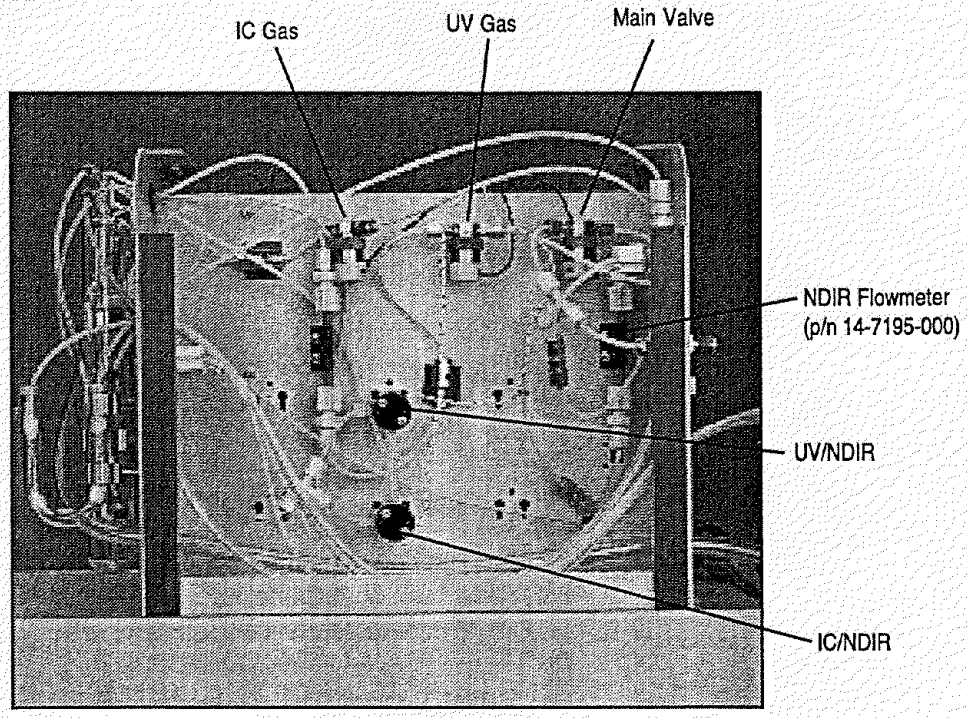


Figure 7-3 Inside View of Right Bay

When a leak is detected outside the unit before the IC sparger:

Locate the IC flowmeter, if installed. If you do not have the IC flowmeter, locate the IC flow restrictor. Remove the inlet tube from the flowmeter (or outlet tube from flow restrictor) and measure the flow. If you obtain a reading of 200 cc/min before the flowmeter (or from a flow restrictor), but you are not receiving 200 cc/min before the IC sparger outside the unit, check the connections at the flowmeter. Replace if necessary.

If you do not obtain a reading of 200 cc/min at the flowmeter (or flow restrictor outlet) inlet, turn the IC valve off. This is done through the software under the **Diagnostics** screen. Remeasure the flowmeter (or flow restrictor) inlet. If you do not obtain a reading of zero, there is a problem with the IC valve. Tighten the fittings on the valve and remeasure. If you still do not obtain a reading of zero, replace the IC valve.

When a leak is detected outside the unit after the IC sparger and before the gas/ liquid separator:

Locate the IC valve and remove the line attched to the common opening. Measure the flow. If you do not obtain a reading of 200 cc/min, check the seal and frit in the IC sparger.

If you obtain a reading of 200 cc/min at the common, remove the blue line to the tee and measure. If you obtain a reading of 200 cc/min, the problem is in the tee. Tighten all tee connections and remeasure. If this does not alleviate the problem, replace the tee.

If you do not obtain a reading of 200 cc/min before the tee connection, the problem is in the IC valve. Locate the IC valve and tighten the fittings. Remeasure the line before the tee connection. If you still do not obtain a reading of 200 cc/min, replace the IC valve.

NOTE: Flow rates for IC must be within 3% of the initial reading.

When a leak is detected outside the unit before the UV reactor:

Locate the UV flowmeter, if installed. If you do not have the UV flowmeter, locate the UV flow restrictor. Remove the inlet tube from the flowmeter (or the outlet from the flow restrictor) and measure. If you obtain a reading of 200 cc/min before the flowmeter (or flow restrictor outlet), but you are not receiving 200 cc/min before the UV reactor outside the unit, check connections and replace flowmeter, if necessary.

If you do not obtain a reading of 200 cc/min at the flowmeter (or flow restrictor outlet) inlet, turn the UV valve off. This is done through the software under the **Diagnostics** screen. Remeasure the flowmeter (or flow restrictor) inlet. If you do not obtain a reading of zero, there is a problem with the UV valve. Tighten the fittings on the valve and remeasure. If you still do not obtain a reading of zero, replace the UV valve.

When a leak is detected outside the unit after the UV reactor and before the gas/ liquid separator:

Locate the UV/NDIR valve and remove the common. Measure the flow. If you do not obtain a reading of 200 cc/min, the problem is with the seal or frit in the UV reactor.

If you obtain a reading of 200 cc/min at the common, remove the blue line to the tee and measure. If you obtain a reading of 200 cc/min, the problem is in the tee. Tighten all tee connections and remeasure. If this does not alleviate the problem, replace the tee.

If you do not obtain a reading of 200 cc/min before the tee connection, the problem is in the UV valve. Locate the UV valve and tighten the fittings. Remeasure the line before the tee connection. If you still do not obtain a reading of 200 cc/min, replace the UV valve.

Fixing a UV Reactor Gas Leak

If the carrier gas leak is traced to the UV Reactor, then follow these steps re-checking the flow after each step:

1. Make sure Teflon Plug is making a good seal with the glass reactor body. Tighten if necessary.
2. Make sure septa are in place at the sparger tube and sample inlets.
3. Make sure sample gas outlet fitting is properly secured in UV Reactor assembly.
4. Make sure screws (2) used to attach metal cap to Teflon plug are providing a good seal for UV Lamp and Sparger tubes. Tighten screws as necessary making sure one screw is not overtightened in relationship to the other. It is important that the metal has a uniform press fit with the Teflon plug.

When a leak is detected outside the unit before the chlorine scrubber:

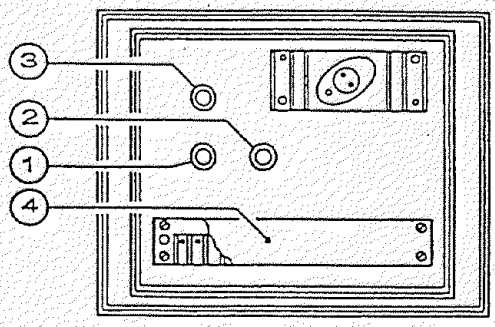
Locate the permeation tube. Remove the green inlet tube and measure the flow with a hand-held flowmeter. If you obtain a reading of 200 cc/min, the problem is in the permeation tube. Tighten all fittings on the permeation tube and remeasure. If you still do not obtain a reading of 200 cc/min, replace the permeation tube (p/n 090-825).

If you obtain a reading of less than 200 cc/min at the permeation tube inlet, the problem is in the mist trap. Check all rubber seals for cracks and make sure they are replaced securely in the mist trap.

When you suspect a leak in the NDIR detector:

Locate the NDIR access panel in the rear of the unit. Remove the four screws. Using Figure 7-3 as a reference, locate the sample gas inlet. Measure this line with a hand-held flowmeter. You should obtain a reading of 200 cc/min. If not, replace the chlorine scrubber.

If you obtain a reading of 200 cc/min at the NDIR inlet, locate the NDIR flowmeter and measure the inlet. If you do not obtain a reading of 200 cc/min at the NDIR flowmeter inlet, the NDIR should be serviced. If you obtain a reading of 200 cc/min at the NDIR flowmeter inlet, locate the loose tube exiting the NDIR flowmeter and measure. If you do not obtain a reading of 200 cc/min here, replace the NDIR flowmeter. If you receive a reading of 200 cc/min here, you have successfully completed the leak checking process.



1. Sample Gas Inlet
2. Sample Gas Outlet
3. Purge Gas Inlet
4. Terminal Block for External Wiring

Figure 7-4 NDIR Rear Panel

7.7 Troubleshooting the Phoenix 8000

When the Phoenix 8000 unexpectedly stops in the middle of an analysis:

User must: reboot the system; empty the IC Sparger, UV Reactor, and Syringe (if necessary); and home the STS 8000 Autosampler.

When the autosampler loses communication with the Phoenix 8000:

User will not receive an error message if such a failure occurs. If a communication failure is suspected (i.e., running event display on run screen stops at an autosampler command), check all STS 8000 Autosampler power and RS-232 connections.

7.8 Calling Tekmar-Dohrmann Service

If you need assistance solving a problem, follow these steps:

1. Write down the model name, model number, and serial number of the instrument.
2. Note the type of problem you are having: write down the conditions under which the problem occurred, and the display, activity, or result that indicated the existence of a problem.
3. Place the manual near the telephone. The service representative may ask you to look at a diagram.
4. Call Tekmar-Dohrmann Service at one of the following numbers:

- (800) 874-2004 toll-free in the US and Canada
- (513) 247-7000 outside the US and Canada

7.9 Returning the Phoenix 8000

Do not return the Phoenix 8000 unless a Tekmar-Dohrmann representative authorizes you to do so. A service representative may be able to help you solve the problem over the telephone. Also, if the instrument must be returned, the representative can tell you how to prevent damage during shipment. **The representative must give you a return authorization number.** Write this number on the return label.

7.10 Spare Parts List

PART #	PART DESCRIPTION
SYRINGE, VALVE,and ASSEMBLIES	
14-7011-052	Syringer drive assembly with 8 port valve and syringer
14-7012-050	Syringe, 8 port valve
14-7127-052	SYRINGE, 25ML W/PLUNGER
14-7040-050	SYRINGE DRIVE for Phoenix 8000
GLASSWARE and ASSEMBLIES in CHEMISTRY AREA	
14-7201-009	Washer, 8 port valve, each
14-7015-024	Reaction vessel, IC, for Phoenix 8000
14-7017-024	Fritted sparge tubes, IC or UV, for Phoenix 8000
14-7020-024	Reaction vessel, UV, for Phoenix 8000
14-7021-024	TFE cap, UV reaction vessel, for Phoenix 8000
14-7183-000	UV-Lamp w/ Pins, Amp Connector and O-Rings, for Phoenix 8000
885-379	Cap, sparger TEF
14-7022-080	Metal cap, UV reaction vessel, for Phoenix 8000
14-7033-043	O-Ring, sparge, in UV reaction vessal for Phoenix 8000
14-7034-043	O-Ring, silicone, UV Lamp, in UV reaction vessel for Phoenix 8000
14-7029-024	Gas/Liquid Separator for Phoenix 8000
050-114	Plug Con 6 CKT Amp 1480704-0
885-143	Mist Trap for Phoenix and DC-180
14-7014-024	Scrubber assembly, chloride, for Phoenix 8000
888-448	Tube, scrubber U shape
511-738	Pyrex wool 1 gram - 1 g
511-876	Tin, Granular, 20 mesh - 1.5 oz.
511-895	Copper, granular, 20 mesh - 1.5 oz.
TUBING, FITTING, SEPTA, and MISCELLANEOUS in CHEMISTRY AREA	
14-7113-002	Tube, TFE 1/16" Red Stripe (price per inch)
14-7114-002	Tube, TFE 1/16" Blue stripe (price per inch)
152-106	Tube, TFE, 1/8" No stripe (price per inch)
14-7108-002	Tube, TFE, 1/8" Red Stripe (price per inch)
14-7109-002	Tube, TFE, 1/8" Black stripe (price per inch)
14-7110-002	Tube, TFE, 1/8" Green stripe price per inch)
14-7111-002	Tube, TFE, 1/8" Yellow stripe (price per inch)
14-7112-002	Tube, TFE, 1/8" Blue stripe (price per inch)
154-424	Tubing, Tygon 1/2" OD (523-359) (price per inch)
517-799 (ea)	Septa connector, silicone, for 1/16" tubing red/white, ea (formerly p/n 517-798, 10 per pack)
517-815	Septa-connector, 1/32" hole, gray, ea (formerly p/n 517-807, 10 per pack)
517-813	Septa-connector, for 1/8" tubing, red, ea (formerly p/n 517-814, 10 per pack)
070-651 (ea)	Septa, solid, silicone, red/white, ea

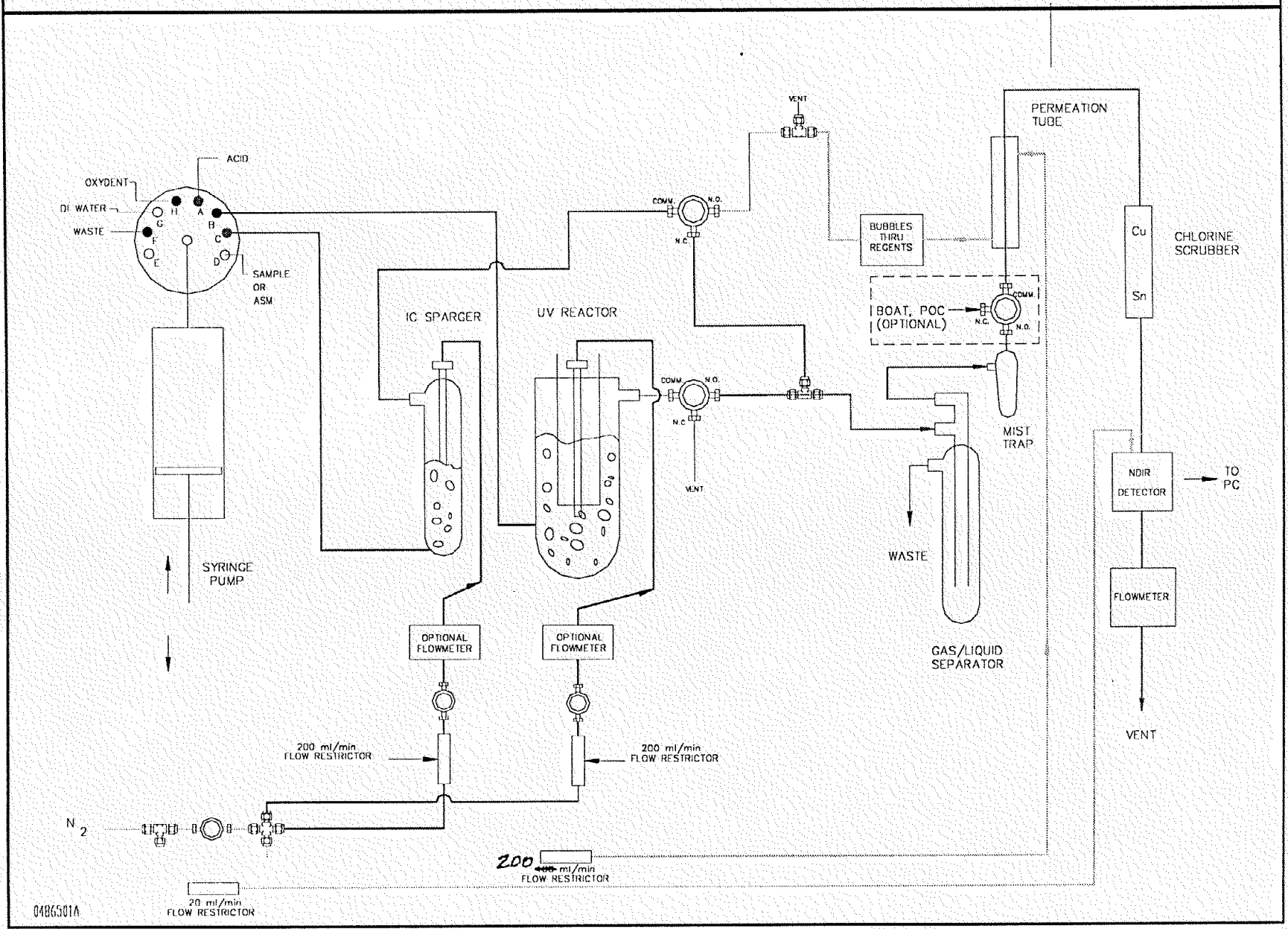
continued

	14-7037-016	FITTING, FLNGLESS, NATURAL 1/8"
	14-7038-016	EXIST BUSHING 1/8 TEFZEL, SHORT
	14-7035-016	FITTING, FLNGLESS, NATURAL 1/16"
	14-7036-016	BUSHING, 1/16" TEFZEL
	14-7013-094	Drip tray for Phoenix 8000
PLUMBING	080-905	CONN 10-32 (M) to 1/8 tube
	080-907	TEE 1/8 UNION POLY
	080-974	REDUCING UNION POLY 1/4T 1/8T
	081-032	20 MICRON, 1/8 SWGLK BRS
	090-093	FLO-RSTCTR 20CC 1/8 UNION YEL
	090-097	FLO-RSTCTR 100CC 1/8 UNION RED
	090-099	FLO-RSTCTR 200 CC 1/8 UNION BLU
	14-7026-050	VALVE, AIR, 24VDC
	14-7025-050	Valve, 2-way air, 24 VDC, for Phoenix 8000
	14-7037-016	FITTING, FLNGLESS, NATURAL 1/8"
	14-7038-016	FITTING, FLNGLESS, NATURAL 1/8"
	090-825	PERMEATION DRYER
	050-490	DIGITAL FLOWMETER, 0-1 L/M 1-5 FDC OUT
	14-4261-016	CROSS, 1/8" UNION, BRASS
DETECTOR	010-905	IR, FUJI 3300 NON-LINEAR, 115V/60HZ
	010-909	IR, FUJI 330 Non-Linear 220V/50HZ
	010-962	Cell liner for Fugi NDIR detector, 125 mm
	073-022	Fuji Cell Window
	073-023	O-Ring, Fuji Cell Window
	14-6860-000	Chopper motor 1 light source Fuji NDIR
ELECTRONICS	14-7009-090	Main CPU board for Phoenix 8000
	14-7008-000	UV Power Supply
	14-7040-050	Syringe and Valve drive
	14-4383-028	Power Module
	14-7007-000	Power Supply

STS 8000 AUTOSAMPLER

14-7252-000	STS 8000 Rear Panel
14-7253-000	STS 8000 Bracket Bar Filler Loop
14-7254-000	STS 8000 Side Cover, left
14-7255-000	STS 8000 Side Cover, right
14-7256-000	STS 8000 Encoder Disk - 28 Tooth
14-7257-000	STS 8000 X Encoder Board Assy
14-7258-000	STS 8000 X Encoder and LED Cable Assy
14-7259-000	STS 8000 X/Y Board Cable Assy
14-7280-000	STS 8000 X Motor Assy
14-7261-000	STS 8000 Main Board Assy
14-7282-000	STS 8000 A/C Power Cable Assy
14-7263-000	STS 8000 D/C Power Cable Assy
14-7264-000	STS 8000 Probe Guide Foot
14-7265-000	STS 8000 .156 Hex Y Drive
14-7266-000	STS 8000 Y End Cap
14-7267-000	STS 8000 Y&Z Board Assy
14-7268-000	STS 8000 Y&Z Motor Assy
14-7269-000	STS 8000 Black Tray
14-7270-000	STS 8000 Z Drive 183mm
14-7271-000	STS 8000 Z Drive 123mm
14-7272-000	STS 8000 Z Drive 56mm
14-7273-000	STS 8000 Tray Spacer Pkg.
14-7274-000	STS 8000 Diverting Valve Assy
14-7275-000	STS 8000 Valve & Connector Assy
14-7276-000	STS 8000 Liquid Level Detector Cable
14-7777-000	STS 8000 Tubing/Support Rod Assy
14-7278-000	STS 8000 Support Bar Assy
14-7279-000	STS 8000 Rack Holder, 301D Series Back
14-7280-000	STS 8000 Rack Holder; 0,7,8,9 Rack
14-7281-000	STS 8000 Shipping Carton
14-7282-000	STS 8000 Insert, Shipping Carton
14-7283-000	STS 8000 Dual 15/12V AC/DC Pwr Supply
14-7284-000	STS 8000 Dual 5/15V AC/DC Pwr Supply
14-7285-000	STS 8000 Terminal Block Conn., 8-pin
14-7286-000	STS 8000 Terminal Clock Conn., 10-pin
14-7287-000	STS 8000 Fuse, 2 amp (250V) T-type

PHOENIX 8000 FLOW DIAGRAM



Understanding Terms and Abbreviations

This section contains definitions for words and abbreviations used in this manual.

Acid — chemical substance used to oxidize inorganic species

Carbon — a naturally abundant nonmetallic element that is in many inorganic and in all organic compounds (C)

Carbon Dioxide — a colorless, odorless noncombustible gas ($C0_2$)

DOC — *dissolved organic carbon*; organic carbon remaining in a sample after the sample has been filtered (usually with a 0.4 micron fitter size)

Inorganic Carbon — carbonate, bicarbonate and dissolved carbon dioxide; a material derived from nonliving, artificial sources

Method — guidelines for analyses

Mode — type of analysis (TC, TOC, IC, TC-IC)

NDIR — *nondispersive infrared detector*; a measuring instrument that provides an electrical signal that is proportional to the concentration of carbon dioxide

NPOC — *nonpurgeable organic carbon*; organic carbon that remains in a sample after the sample has been purged with gas; commonly referred to as TOC

Organic Carbon — a material that is derived from decaying vegetation, bacterial growth, metabolic activities of living organisms or chemical waste

Oxidation — a reaction in which an element's capability to unite with other substances is increased due to the element's loss of electrons (subatomic particles)

Parameter — a specification or guideline, such as time or temperature

Persulfate — ($S_2O_8^{2-}$) and oxidation agent used to convert inorganic carbon into carbon dioxide

POC	*purgeable organic carbon*; organic carbon that has been sparged or removed from a sample
Purge	to sparge; to allow gas to flow through a sample to remove volatile elements in the sample
Reaction Time	time in which the sample (without the aid of nitrogen flow) reacts with reagent
Reagent	substance used in a chemical reaction to interact and produce
SOC	*suspended organic carbon*; also called *particulate organic carbon*; the organic carbon in particles that are too large to pass through a filter
Sparge	see *purge*
Standard	a sample that contains a known amount of carbon; analyzed for calibration to maintain precision and accuracy
TC	total carbon; all the carbon in the sample, including both inorganic and organic carbon
TIC	total inorganic carbon; also see *inorganic carbon*
TOC	total organic carbon; see also *organic carbon*

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