

# StarLab User Guide



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Service Support: http://www.tocdirect.com

#### **StarLab Operation Manual**

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# Part

#### 1 Introduction

#### 1.1 Important Information

Here are some important details which the user needs to be aware of. These are to ensure safety and reliability.

- All of our components are factory-certified and in most cases made expressly for our analyzers.
   Use of third-party components not recommended by Star could result in warranty invalidation and decreased reliability.
- Before trying to initiate gas flow through the analyzer, it must be connected to a source of dry CO<sub>2</sub>free instrument air. Otherwise, the mass flow controller could become damaged, and would not be
  covered by your warranty.
- When servicing any components, the analyzer should be shut down and unplugged from all sources
  of voltage. This is to ensure user safety, and to help protect the other components.
- When servicing the high temperature reactor/solids module, the unit should be shut down, disconnected, and be given adequate time to cool (at least 2 hours). These components operate at extremely high temperatures at their cores, and ittake a while to dissipate the heat.
- Operators should keep hands/jewelry, etc. out of the motion paths of the autosampler while it is operating. For safety, the plastic shield should be used when the analyzer is operated.
- Care should be used when handling liquids around any electrical equipment. It is recommended
  that the analyzer and any accompanying modules be plugged into a certified GFCI outlet for
  additional protection.
- It is possible that under some circumstances (depending on the sample species), small amounts of toxic gases may be produced during the oxidation process. It is important that the analyzer is properly vented, per installation instructions, Part III.
- The light source used in the STARLAB Analyzer consists of a UV Lamp. Avoid direct prolonged viewing of the bare lamp when it is lit, as it could cause eye injury. It is safe to view the lamp for operation if done indirectly or through glass tubing or protective eye ware.

#### 1.2 Introduction

Thank you for selecting the **StarLab Automatic TOC Analyzer** for your application. We pledge our complete, professional support to you and your application.

This manual includes all necessary information to help you operate the custom StarLab software, designed for your unit.

The StarLab software suite was designed to make laboratory TOC, TC, TIC analysis easy, eliminating many of the irratations of alternate analyzers. It is built on he robust Windows 32-bit platform, to provide maximum performance and compatibility. If there are any questions which you feel are unanswered by the help system, please consult our on-line service at <a href="https://www.starinstruments.com">www.starinstruments.com</a>. Each analyzer is shipped with a high quality webcam, which may be used for real-time personal service and operational support.

### 1.3 Requirements

The minimum equirements for StarLab software are as follows:

IBM Compatible PC

- At least 1 GHz Pentium Processor
- 256 MB of RAM
- Windows XP Professional
- 150 MB of free hard disk space
- 1 free RS-232 serial port\*
- 1024 x 768 screen resolution

\*PCs not including an onboard RS-232 serial port may attempt to use a USB converter. However, this configuration will not be supported. It is suggested that the host PC have a hardware serial port onboard.

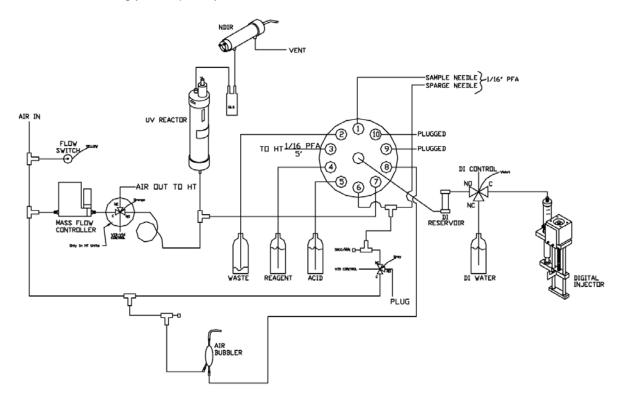
In addition, a printer may be used to print data. However this is not a requirement.

# Part III

#### 2 General Information

#### 2.1 Measurement Principles and Flow Diagram

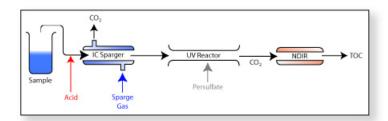
The general flow diagram is shown below. Note that this standard configuration is capable of handling a growing number of analyses. The design architecture of the StarLab also allows for future upgrades to added, extending your capability.



The StarLab analyzer is capable of TOC, TIC, and TC analysis using the following methods:

- Heated Persulfate
- <u>UV/Heated Persulfate</u>
- High Temperature Combustion 9
- Ozone Promoted
- Ultra-Pure

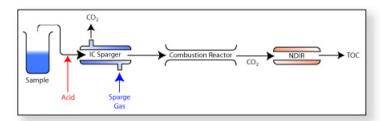
#### 2.1.1 UV/Heated Persulfate Oxidation



Sample is mixed with acid, lowering the pH to 2.0. This converts the inorganic carbon to dissolved CO<sub>2</sub>, which is Air/O<sub>2</sub> stripped out of the solution. Persulfate reagent is added and the remaining organic carbon is then oxidized in the Reactor to form CO<sub>2</sub>, which is detected by the NDIR as a direct

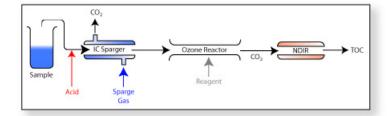
correlation to TOC.

#### 2.1.2 High Temperature Combustion Oxidation



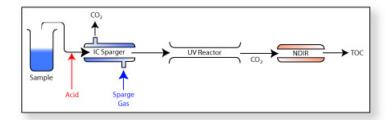
Sample is mixed with acid, lowering the pH to 2.0. This converts the inorganic carbon to dissolved CO<sub>2</sub>, which is Air/O<sub>2</sub> stripped out of the solution. The carbonate-free sample is then directed into the combustion reactor where the remaining organic carbon is then oxidized at high temperature to CO<sub>2</sub>, which is detected by the NDIR as a direct correlation to TOC.

#### 2.1.3 Ozone Promoted Oxidation



Sample is mixed with acid, lowering the pH to 2.0. This converts the inorganic carbon to dissolved  $CO_2$ , which is  $Air/O_2$  stripped out of the solution. Reagent is added and the remaining organic carbon is oxidized by ozone/hydroxyl radicals to  $CO_2$ , which is detected by the NDIR as a direct correlation to TOC.

#### 2.1.4 Ultra-Pure Oxidation



Sample is mixed with acid, lowering the pH to 2.0. This converts the inorganic carbon to dissolved  $CO_2$ , which is Air/ $O_2$  stripped out of the solution. The remaining organic carbon is then oxidized in the UV reactor to  $CO_2$ , which is detected by the NDIR as a direct correlation to TOC.

# Part IIII

### 3 Chapter 2

#### 3.1 Connecting the Analyzer

The StarLab Analyzer requires connection to the PC, a source of dry CO<sub>2</sub> and hydrocarbon-free instrument air or oxygen and liquid/reagent connections. Those connections are detailed in this chapter.

#### 3.1.1 Reagents

Depending upon your application, up to 5 reagent lines may need to be connected. All liquid/gas connections are located on the left side of the unit, and all electrical connections are on the right side of the unit.

If you are using the High Temperature Combustion method with acidification, you should use sulfuric acid. For other methods, phosphoric acid is preferred.

NOTE: DO NOT USE PHOSPHORIC ACID WITH HIGH TEMPERATURE COMBUSTION.

The DI Water line should be connected to a clean source of DI water.

The Air line is to be connected to a source of dry, CO<sub>2</sub> and hydrocarbon-free instrument air or oxygen with a stable pressure of +/-15 psi.

There are 3 drain lines. The larger 1/2" lines (one on the bottom left of the analyzer, one coming from the autosampler tray) should be routed to a gravity-fed drain at a downward slope. The smaller 1/8" line is a pressurized drain and can be fed either to the same gravity drain as the other two, or to a separate container, but it does not need a downward slope.

#### 3.1.2 Communications

With the PC and analyzer both turned off, locate the DB-9 connector on the top left side of the analyzer. This should be connected to a free RS-232 serial port on the PC via a straight-through cable. After connecting, turn on your analyzer, then the PC.



### 3.2 Initial Software Setup

If your computer was bundled with your analyzer, then this section may be skipped, as it is preconfigured from the factory for you.

To install StarLab, insert your CD-ROM into a drive. If the Auto-Run menu does not start, then navigate to 'My Computer', then the drive with the inserted disc.

In this folder, double-click 'Setup.exe'. This will launch the installation program. It is suggested that you allow the software to install to the default locations. Once complete, you must reboot your computer.

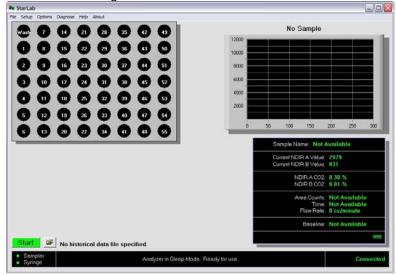
Upon starting the software for the first time after installing, you will be told that the configuration file is corrupt and all default settings will be loaded. The program will then shut down, and must be restarted to use the default settings. When prompted to connect to the analyzer, only select 'Yes' if the analyzer is connected to port COM1 on your PC and turned on. Otherwise, a communication error will occur.

If your analyzer is not on COM1, then select 'No'. This will start the program in Off-Line mode.

Off-Line mode can be determined by the 'Not Connected' message located at the bottom right of the screen.

To set the COM port, select the 'Options' menu item, and then the 'Configure' selection.

Here, you can set the default results path, and the COM port. Once the configuration is finished, press the 'Save' button to save your changes. The program will shut down, and when restarted, will use the new configuration.



# Part

### 4 Chapter 3

#### 4.1 Getting Started

If the software has been configured properly either at the factory, or using the instructions provided in 'Initial Setup' (12), the analyzer may be brought on-line.

When starting the software select 'Yes' when prompted to connect to the analyzer.



#### 4.2 The Main Run Screen

Upon connecting to the analyzer, you will be presented with the main status screen. This screen contains important information about your analyzer, as well as reflecting results during sample runs.

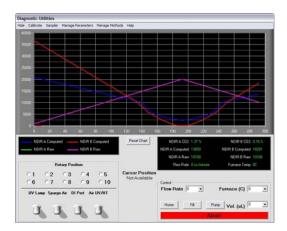


The dark area to the bottom right of the screen is the real-time status window. This reflects the current status of the NDIR sensors, the calculated CO<sub>2</sub> percentages, and real-time data as samples are being run. The number at the bottom right of this window is the analyzer firmware revision.

The bar at the bottom of the screen is the general status bar. To the right, there is an area which gives the busy status for the auto-sampler and the syringe. In the middle, any important information is updated. During the course of a sample run, each step is reflected in this area. The right side provides the connection status.

# 4.3 The Diagnostic Screen

The StarLab software has very robust and powerful diagnostic utilities. Some of these can be used during sample runs to get a better idea for how the analyzer is functioning. To access these tools, click 'Diagnose' (ALT-D) at the top of the Main Run Screen.

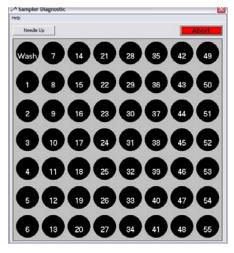


The controls on this screen can be used to maually manipulate the valves, pump, and gas flow rate. These controls are disabled when a sample run has been initiated. The chart display at the top of the screen represents the NDIR sensor signals. There is a real-time status window below to the right. A screenshot of this window can be printed to your default printer by simply pressing 'Print' on the menu bar.

This screen can be hidden at any time by pressing 'Hide' on the menu bar at the top.

#### 4.4 The Sampler Screen

To manipulate the auto-sampler, simply open the Sampler screen by pressing 'Sampler' on the menu bar.



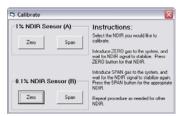
This will allow you to move the auto-sampler to any position by simply clicking the station you choose. While the sampler is busy moving, all stations will be disabled.

### 4.5 Calibrating the NDIR Sensors

Before initial use of your analyzer, you should calibrate the NDIR sensors. This will ensure the most accurate results, and it is not a complicated procedure. To begin, go the Diagnostic Screen. Locate the Flow Rate setting, and click it to select 200 cc/min (it is important that you have instrument air or oxygen connected to the analyzer anytime you initiate gas flow).

After a few seconds, the information panel will reflect the new flow rate. Watch the NDIR graph until both NDIR sensors stabilize with little to no deviation.

To begin calibration, select 'Calibrate' (ALT-C) on the menu bar.



This will open the NDIR calibration screen.

Once both NDIR signals are stable, you may zero the analyzer using you instrument air. Press 'Zero' first for NDIR A. Once the 30 second procedure has completed, then you may do the same for NDIR B.

After NDIR B is finished performing the zero procedure, open the StarLab cabinet using the supplied key. The NDIR sensors are located at the top of the cabinet. Referencing figure \_\_\_\_, disconnect the main gas supply line from NDIR A and connect a bottle of 1.0% CO<sub>2</sub> calibration standard gas to the inlet. Turn on gas flow and watch the NDIR graph on the Diagnostic Screen change.

After a few minutes, the signal will normalize with very little deviation. Once this has occured, press the 'Span' button under 1% Sensor on the Calibrate Screen.

After 30 seconds, the new calibration will be completed. Disconnect the 1% gas and connect a 0.1% standard to complete calibration of NDIR B. This is done indentiacal to NDIR A, except you will use the 0.1% NDIR section of the Calibration Screen.

Once you are finished, close the screen using the button in the top right corner, where you will be prompted to save your calibration. If at anytime you believe that the calibration was faulty, you may say 'No' and the old calibration will be reinstated. However, once you have saved a calibration the previous one is lost.

This completes your calibration. You are now ready to configure your samples.

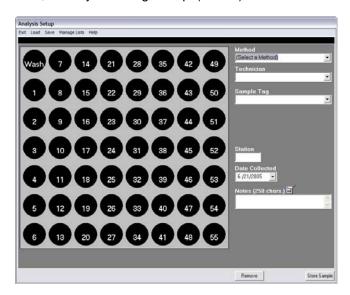
# Part

## 5 Chapter 4

#### 5.1 Setting Up a Sample Run

We will set up a simple sample run consisting of one 'Span Calibration' and one sample.

First, start by selecting 'Setup' (ALT-S) on the Main Run Screen menu bar.



To begin, select sample station 1. Now, you must select a method in the drop-down box to the upper right.



You must enter the technician's name in the approriate box. This will be used for record-keeping. A list of frequently used technician names may be created and accessed via the pull-down arrow to the side. (See Managing Lists 21)

If your sample has a unique name associated with it, enter it in the 'Sample Tag' box. This also has a list function for frequently used values. (See Managing Lists [21])

If this sample is calibration standard, select the 'Calibration Standard' checkbox. Now, select the calibration value (we will use 100 for our example).

The station box is automatically filled in, and cannot be changed. If your sample was collected on a certain day, you may reflect this by selecting the day on the 'Date Collected' drop-down box. Otherwise, leave it on its default value of the current day.

Any notes which are associated with your sample may be entered in the 'Notes' box.

Store this sample by pressing the Store Sample button.

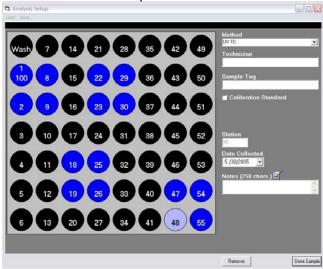
Repeat this procedure for station 2, except do not click the Calibration Standard box, as we will assume station 2 is our sample.

In most cases, you would simply click the station which is a sample, and then click

the Store Sample button. However, any notes or collection date may be desired.

In a real live sample run, you would do this for as many samples as you have. Keep in mind that all stations do not need to be run sequentially, so you could load the tray in groups. This may assist in helping to keep operators from being confused as to which samples came from where.

This would be an example:



Once you are finished configuring the sample run, you may save your analysis job by selecting "SAVE" on the menu bar or exit the screen and return to the Main Run screen.

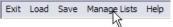
Your sample runs may be saved and loaded later for convenience, or to repeat a job which does not change very often. This is useful in laboratories that monitor the same samples continually. To save a job, simply click 'Save'. To load a previously created job, click 'Load'.

Exit Load Save Manage Lists Help

# 5.2 Managing Lists

Lists of frequently used values can save time in setting up a sample run. These lists are available for two designations: Technician Name and Sample Tag.

To set up/manage your lists, click on 'Manage Lists' in the menu bar on the Setup screen.



The Manage Lists screen will appear.



You may add or edit names by using the text entry boxes, then pressing the \_\_\_\_\_\_ button to the side.

Names may be deleted by highlighting them, then pressing Delete User or Delete Tag or The list changes will not be effective until they are saved using the save option in the top menu.

#### 5.3 Starting the Analysis

Once all samples have been configured, you may begin the analysis on the Main Run screen. Before starting you may specify a results file to store the analysis results by pressing the button in the bottom left corner of the screen. If you choose not to do this, a unique file name will be generated automatically. This ensures that no previous data can be lost due to error.

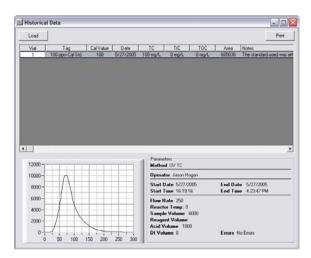
While the analyzer is performing an analysis, a number of useful things are indicated on this screen. At the bottom center of the screen, every step is communicated to the user. Also in this area is the sampler and syringe status lights.

When running, the sensor graph in the top right will show the NDIR values as they are integrated by the computer.

#### 5.4 Retrieving Stored Results

After a sample run has been completed, you may retrieve the results at any time in the future by going to the Viewer screen on the menu bar of the Main Run screen (ALT-V).

Once in this screen, click the Load button to select a results file to view.



In the list view at the top of the screen, all sample data are shown for every sample from that run. You may get a detailed view of the sample by highlighting the row that the sample is located on. This will recall the actual sensor signal. A simple list of the samples and results may be printed by pressing the

button. A more detailed view of any individual sample may be printed by highlighting the sample and right-clicking to select 'Print Sample Detail'. This view is very comprehensive and

includes data regarding the analysis parameters, as well as the sensor signal.

Also indicated on the screen is the error status of the run. If a job is aborted, all results up until the abort are stored. Thus, you can see if a job was executed to completion by checking the 'Errors' field in the bottom right section of the screen.

# Part

## 6 Chapter 5

This chapter will cover basic analyzer maintenance, parts exchange procedures, and how to make necessary standards/reagents. Where ratio charts are presented, it is suggested that you print these and make them available where operators and technicians can view them while preparing their liquids.

#### 6.1 Standards

There are many chemical standards to choose from. For most applications, Star recommends KHP or Ethylene Glycol for carbon, and ammonium sulfate for nitrogen. Tables detailing ALL of the available standards are included, but the two mentioned previusly will usually be sufficient.

Star also offers high-purity standards as well as reagents through either a subscription service, or asneeded. For more information, contact us 4.

#### **6.1.1** Carbon

	100 mg/Liter TOC (AS CARBON)	1000
mg/Liter TOC (AS CARBON)		
Ethylana Ohyaal	0001/1 11-0	0.001/1.11-0
Ethylene Glycol	.233 ml/L H <sub>2</sub> 0	2.33 ml/L H <sub>2</sub> 0
Methanol	.337 ml/L H <sub>2</sub> 0	3.37 ml/L H <sub>2</sub> 0
Ethanol	.242 ml/L H <sub>2</sub> 0	$2.42 \text{ ml/L H}_{2}^{-}0$
Acetone	.204 ml/L H <sub>2</sub> 0	$2.04 \text{ ml/L H}_{2}^{-}0$
Carbon Tetrachloride	.807 ml/L H <sub>2</sub> 0	8.07 ml/L $H_2^-$ 0
Sucrose	.238 gm/L H <sub>2</sub> 0	2.38 gm/L H <sub>2</sub> 0
Urea	.500 gm/L H <sub>2</sub> 0	5.00 gm/L H <sub>2</sub> 0
Acetic Acid	.250 gm/L H <sub>2</sub> 0	2.50 gm/L H <sub>2</sub> 0
KHP (Potassium acid phthalate)	.212 gm/L H <sub>2</sub> 0	2.12 gm/L H <sub>2</sub> 0
Glycine	.313 gm/L H <sub>2</sub> 0	3.13 gm/L H <sub>2</sub> 0
Sodium Stearate	.141 gm/L H <sub>2</sub> 0	1.41 gm/L H <sub>2</sub> 0
Succinic Acid	.246 gm/L H <sub>2</sub> 0	2.46 gm/L H <sub>2</sub> 0
Sodium Oxalate	.555 gm/L H <sub>2</sub> 0	5.55 gm/L H <sub>2</sub> 0

Add amounts shown (in milliliters or grams) to clean, dry, one liter volumetric flask and dilute to one (1) liter with distilled, deionized water. Lower mg/L values are obtained by dilution.

EG (Ethylene Glycol) or KHP (Potassium Acid Phthalate) are recommended for the TOC Calibration standard solution.

Example of making a TOC standard:

The customers sample stream normally runs a TOC Value of no higher than 2000 ppm.

To prepare a 2000 ppm span solution from Ethylene Glycol consult the Preparation of Standards Chart. You will see it takes 2.33mLs of Ethylene Glycol into 1 liter of DI to make a 1000 ppm standard.

$$2.33 \text{ m/s } x 2 = 4.66 \text{ m/s}$$

Add 4.66 mls of Ethylene Glycol into 1 Liter of DI water to make a 2000 ppm span solution.

#### 6.2 Reagents

The proper methods for creating reagents are listed in this chapter. It is very important that the DI water used be as clean as possible, especially in low carbon ranges. Also, all reagent containers used should be thoroughly rinsed with DI water, so as not to contaminate your sample.

#### 6.2.1 Sodium Persulfate

Sodium persulfate is a strong oxidizing agent. Take all necessary preautions as when handling any oxidizing compound.

- 1. All chemicals, including water, should be reagent grade or better and free of carbon-containing compounds.
- 2. The container used should be flushed with distilled or deionized (DI) water.
- 3. Dissolve **238 grams** of ultra-pure reagent grade sodium persulfate (Star P/N 40000) into **one (1)** liter of D.I. water.
- 4. Do not heat reagent to dissolve, as this is not necessary and can reduce the effectiveness of the solution.
- 5. Insert tubing from the Persulfate port on the analyzer into the persulfate container.

It should be noted that some "reagent grade" sodium persulfate is not really "pure". All persulfate reagent supplied by Star is guaranteed to have sufficient purity for proper operation.

#### 6.2.2 Acid

Two types of acid may be used with the StarLab analyzer:

- Sulfuric Acid [27]
- Phosphoric Acid 27

Generally, phosphoric acid is recommended for use with Heated Persulfate and any UV-based methods. All combustion-based methods must use sulfuric acid. It is NOT recommended to use phosphoric in you high temperature combustion reactor. Doing so will result premature catalyst destruction and blockage of the orifices.

#### 6.2.2.1 Phosphoric Acid

Note: Phosphoric acid should NOT to be used with high temperature combustion methods.

- 1. The container used should be flushed with distilled or deionized (D.I.) water.
- 2. Partially fill container with DI water.
- 3. Add 117ml of 85% phosphoric acid into the partially filled container.
- 4. Dilute with additional DI water to 1 liter.

#### 6.2.2.2 Sulfuric Acid

Sulfuric acid is used for combustion-based methods. Phosphoric acid should not be used.

To prepare sulfuric acid:

- Flush container to be used with DI water thoroughly
- Add 56mL of 66 baume sulfuric acid to 1 litre of DI water
- Recommended reagent container size is 1 litre
- For safety, the reagent container should be labeled "ACID"

# Part VIII

# 7 Chapter 6

### 7.1 Parts

Enter topic text here.

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