

# IRGAS-XSA

(IRGAS Moisture + Plus)

## User's Guide

Version 3.6

December 2006



***CIC Photonics***

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 Name: Richard T. Meyer  
 Title: CEO & President  
 Date: \_\_\_\_\_

By: \_\_\_\_\_  
 Signed: \_\_\_\_\_  
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## 1.0 Introduction

The IRGAS-XSA Turnkey Gas Analysis System is a laboratory version of CIC Photonics, Inc.'s product line of IRGAS – Integrated Real-Time Gas Analysis Solutions. The IRGAS line has been designed and developed to serve the requirements of the gas industry. Typically this entails the measurement of gas compositions within carrier gases at the low ppm level within seconds.

The IRGAS products are designed for service with the numerous corrosive and toxic gases that are used in the semiconductor and electronic specialty gas industries. The heart of the IRGAS is the nickel-plated stainless steel Ranger-EN™ long path gas cell; it provides the direct interface with the reactant gas flows.

The IRGAS is an industrial turnkey system that provides an automatic on-line monitoring analytical tool for the measurement of gas compositions in real-time. The system is based on FTIR Spectroscopy and controlled by SPGAS (Specialty Gas Analysis Software), which is a sophisticated software running on a personal computer. A system architecture was designed to provide the integration and control of the hardware elements, as well as the spectral analysis and chemometrics, to determine the composition of gas streams (see Figure 1).

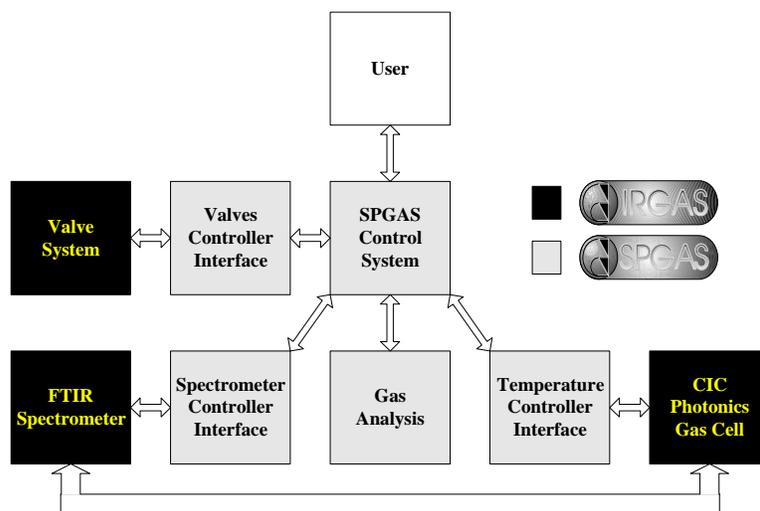
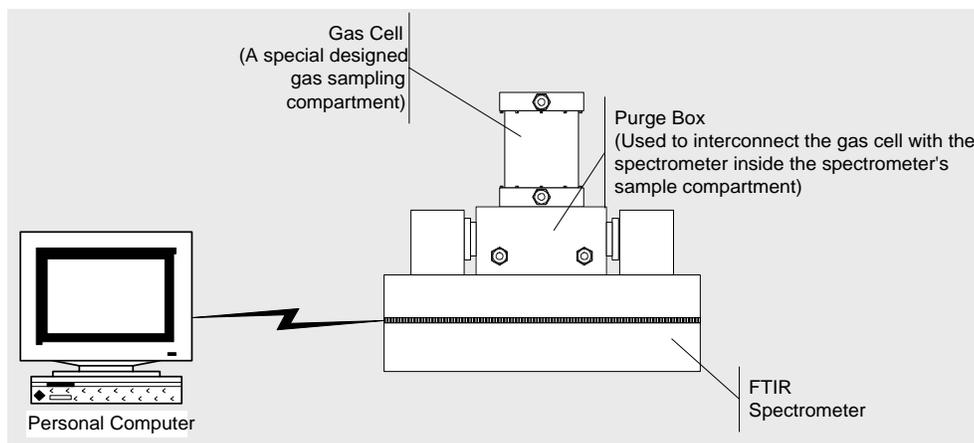


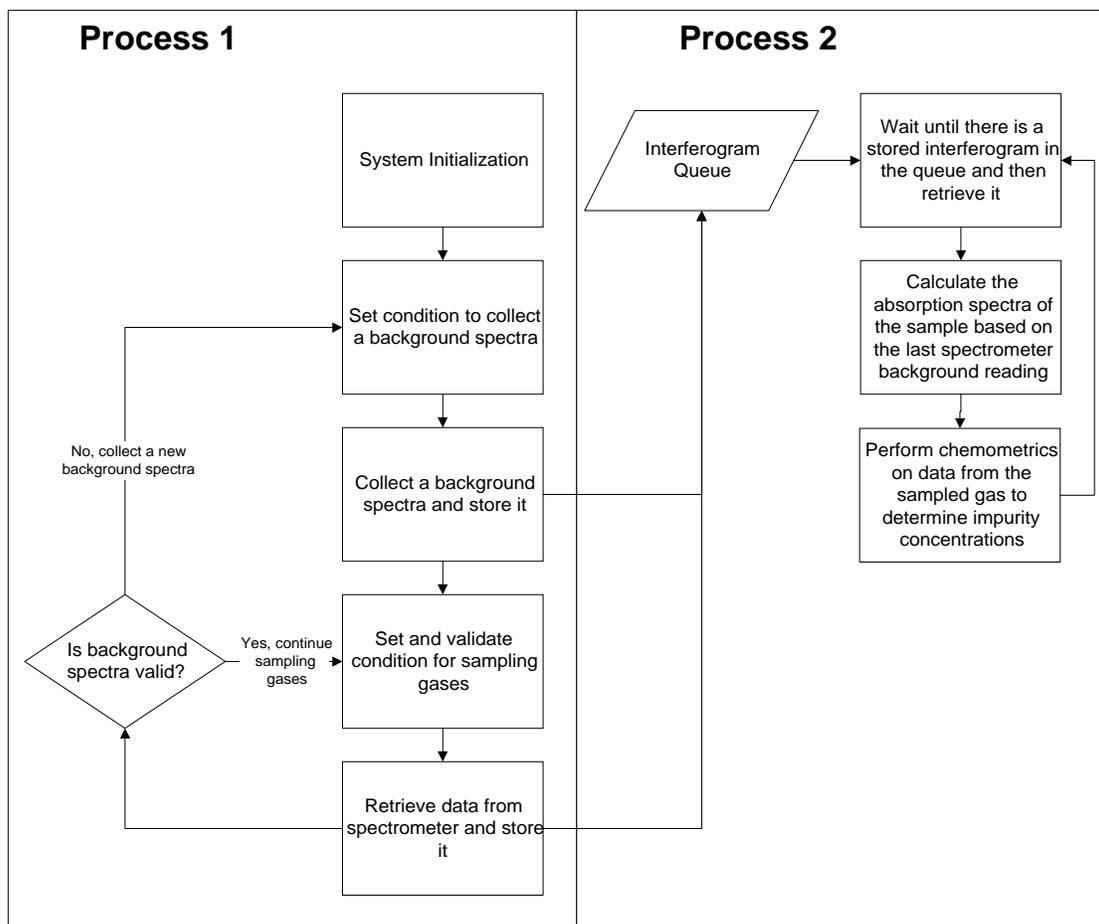
Figure 1 System Block Diagram

FTIR Spectroscopy offers the best characteristics to perform real-time measurement of gas compositions. Since the infrared light detector is not in contact with the corrosive sample, it is not affected and the instrument can, therefore, be set for a continuous operation and scanning of the sample.

The real-time gas analysis is performed through an FTIR Spectrometer, which contains a special gas sample compartment (*gas cell*) that is connected directly to a gas line that supplies the sample gas. Since the spectrometer is connected directly to a computer (see Figure 2), the spectrometer can be set to perform continuously an x number of scans of the sample gas and store this data as an interferogram. Every time an interferogram is completed, the computer retrieves this interferogram from the spectrometer and performs the quantitative gas analysis of the impurities.



**Figure 2 FTIR Spectrometer and Personal Computer Interconnection**



**Figure 3 Processes 1 and 2 block diagram**

The gas analysis process is divided in two main processes (Figure 3). One process is in charge of the data collection and the second process is in charge of the data processing.

Process 1 controls the interaction between the spectrometer and the computer. Additionally, Process 1 interacts with and synchronizes other sub-processes that set and control the sampling and

the environmental conditions of the spectrometer. These sub-processes are in charge of controlling the gas handling system and the temperature controller if required.

Performance data for the IRGAS can be found on the web site [www.irgas.com](http://www.irgas.com), and in technical papers that have been presented at IFPAC, Pittcon, and SemiconWest conferences and expositions.

## **2.0 Unpacking and Installation**

The IRGAS-XSA is a high performance instrument that includes an interferometer, laser, mirrors, windows, filters, high vacuum seals, and electronics. Hence, it must always be handled with a degree of care and precaution to prevent physical damage and optical misalignments. The IRGAS-XSA has been packed within two heavy-wall cardboard boxes for shipping.

### **2.1 Removal from Boxes**

The IRGAS-XSA has been shipped inside two cardboard boxes, with foam and packing materials between the two boxes and with loose foam surrounding the instrument itself; the instrument is enclosed within a plastic bag to keep it dry and clean.

The total weight of the IRGAS-XSA instrument is approximately 150 lbs.

### **2.2 Electrical, Electronic, and Gas Connections**

The IRGAS-XSA instrument consists of a WORKIR FTPA2000-300 FTIR spectrometer, on top of which is mounted the heated Ranger-EN gas cell, temperature controller, and a manifold-bracket upon which are mounted the valves and heater controls for the ¼” stainless steel sample and exhaust plumbing lines.

## **3.0 Description of Major Components**

### **3.1 IRGAS System Overview**

A System Block Diagram is provided in Figure 1. As will be seen, the use and operation of the IRGAS System are in general quite simple and easy to perform. IRGAS offers a robust design and works under Microsoft's Windows NT/2000/XP Operating System. The minimum requirements of the system are as follow:

IRGAS-XSA Minimum Hardware Requirements:

- Pentium or AMD 400 Mhz or better
- 128 MB Ram
- One free Serial Port
- 1 GB of free hard drive space for data storage
- Ethernet 10/100 Base T network adapter

Software Requirements:

- Windows NT/2000/XP for Ethernet communications

### 3.1.1 Installation

Insert the software CD into your computer's CD-ROM drive. Your computer should automatically read the disk and display the installation screen. If the installation screen does not appear automatically, double click the 'My Computer' icon to open it, double click on the CD-ROM drive where you placed the installation CD, search for the application called Setup.exe and double click on it to launch the installation program.

Follow the on-screen instructions to install the software. After the software has been installed, the software needs to be activated.

### 3.1.1 Activation

Not applicable.

## 3.2 IRGAS System Operational Modes

The IRGAS system takes advantage of the built-in security features that Windows NT/2000/XP offers. These features allow one to set an account that will run the system in Operational Mode and another account for system Maintenance Mode.

By logging-on in the Operational account, the IRGAS system starts automatically (see Figure 4), and when the IRGAS system is closed, it automatically logs-off from the Operational account session.

The Maintenance account for the IRGAS system allows access to all the IRGAS system configuration features. The IRGAS system does not start automatically every time the user logs-on in the Maintenance account. The Maintenance account provides access to the Windows NT standard desktop, from which the IRGAS system can be accessed or updated. The user can run the IRGAS System by double clicking on the IRGAS icon on the Window's screen.

### 3.3 Bomem WORKIR FTPA2000-300 FTIR Spectrometer

The Bomem WORKIR FTPA2000-300 FTIR spectrometer was chosen for the IRGAS-XSA because of its high quality patented interferometer, its total compactness and modest weight, and its industrial ruggedness. Technical features of the WORKIR FTPA2000-300 are provided on the next page. The internal optics are KBr and the IR detector is a TE-InAs detector. In addition to this, is a TE Cooled Power Supply. Once the user has begun purging the system turn on the TE Cooled Power Supply. **The TE Cooled Power Supply should not be turned on without first starting to purge the system.** The WORKIR FTPA2000-300 internal compartment should be purged with ultra-dry nitrogen at all times. Electrical power is provided to the WORKIR FTPA2000-300 when the "System" switch is turned on; it should be left "on" at all times to keep the spectrometer at a stable operating temperature and to reduce ambient moisture collection on the hygroscopic KBr optics; the cooling fans are continuously operating during this time. If the IRGAS-XSA is "stored" for any period of time, the WORKIR FTPA2000-300 should first be purged and sealed with dry nitrogen. A complete Instruction Manual for the WORKIR FTPA2000-300 is provided.

### 3.4 Ranger-EN Long Path Gas Cell

The Ranger-EN Long Path Gas Cell is a nickel-coated aluminum cell with gold-coated mirrors and a cylindrical heater. The cell is sealed with Kalrez O-rings. The IR beam input/output windows are KBr windows. The window housing is equipped with gas connectors for purging.

The WORKIR FTPA2000-300, its optical housings, and the Ranger-EN window housing should be continuously purged with ultra-dry nitrogen at all times.

The Ranger-EN gas cell volume should also be continuously purged when not being used to measure a process gas stream.

See also the User Manual for the Ranger-EN gas cell.

### 3.5 Temperature Controller

The Ranger-EN cell body is controlled with a 1/16 DIN Microprocessor-Based Auto-Tuning Controller. It drives a sheet heater with an imbedded Type K thermocouple that is mounted cylindrically inside the heater assembly. The temperature setting is manually adjustable with touch switches on the face of the Controller. The preset readout is in °C. Normal operating temperature range is ambient to 200°C. The Kalrez O-rings are rated to 300°C for continuous use.

**Always operate the Temperature Controller in the “auto-tune mode” with a preset temperature maximum; that ensures that the temperature does not exceed the safe limit for the heater and the O-rings.**

An instruction manual for the Watlow controller is provided.

### 3.6 CPU and Monitor

To be provided by the end user.

### 3.7 Purge and Sample Gas Plumbing

Swagelok ball valves and ¼” Swagelok connections for the process gas (in and out) are provided on the manifold-bracket that is mounted on the backside of the FTIR . A particle filter is mounted at the front end of the sample gas line.

The user must provide all mass flow controllers and other devices that may be required for handling the process gas. In addition, a separate connection to a house-supply of purified ultra-dry nitrogen is required for purging the Ranger-EN gas cell between process gas samples and for recording background spectra.

### 3.8 SPGAS System Management and Gas Quantification Software

The complete version of CIC Photonics' proprietary software, SPGAS, provides both system management of the hardware components and advanced gas quantification capability. The version installed in the IRGAS-XSA requires some manual operations of the hardware components—e.g. setting the temperature controller, opening/closing gas valves, etc.

**The SPGAS software for this specific IRGAS-XSA has real-gas generated calibration data preinstalled for H<sub>2</sub>O in NH<sub>3</sub> only. The calibration is for a temperature of 60°C and a pressure of 1.1 atm absolute (836 torr absolute).**

### 3.9 Electrical System

The electrical system for the IRGAS system is organized around a plug strip with a 15 amp load rating. There are two A/C loads: the Bomem FTIR Spectrometer and the temperature controller/heater.

## 4.0 System Start-up Operations

### 4.1 Purge Gas Flow and System Switches

First start the flow of the ultra-dry nitrogen purge gas through the spectrometer and transfer mirror optics and separately through the gas cell; then turn on the System and Computer switches.

Purging of the system requires a total of 10 slpm; 5 slpm for the spectrometer and 5 slpm for the optical coupling assembly. These purge rates are achieved by providing 30 psig at the entrance of the system purge valve.

In addition and separately, the gas cell needs to be purged before and after any flow of process gases. This is best achieved by using the 3-way valve to switch from the sample gas to the N<sub>2</sub> supply.

### 4.2 Temperature Controller and Gas Cell Heater

We recommend using 60°C as the on-going operational temperature for the gas cell during gas analyses and 125°C for the bakeout periods.

The Watlow temperature controller has been preset at the factory at 60°C; it is manually adjustable to other temperatures.

For the initial bakeout of the gas cell, we recommend 125°C with purge nitrogen for a 24-hour start-up conditioning period.

### 4.3 FTIR Spectrometer

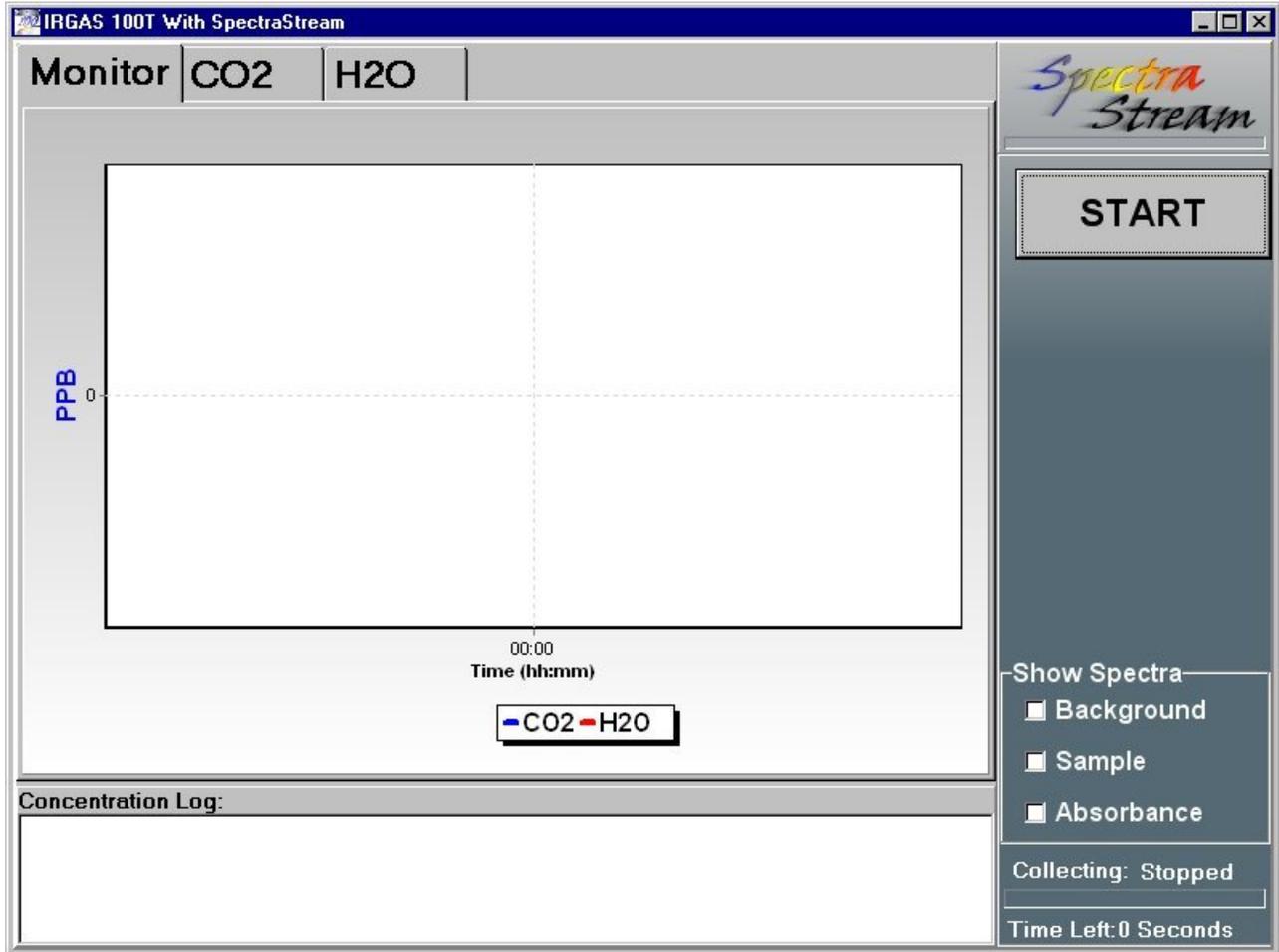
The FTIR spectrometer is turned on when the System switch is turned on. It requires between three to six hours to stabilize for quantitative data collection; but it does start operating as soon as the System switch is turned on. The FTIR resolution has been preset at the factory for 2 cm<sup>-1</sup>, which is the resolution required for use with the SPGAS software; **therefore, do not make any attempt to change the resolution.**

### 4.4 Computer System

The end user's CPU and Monitor become activated when its switch is turned on and the IRGAS-XSA program is loaded. The first screen to appear will look like Figure 4. Toward the middle upper right of the screen is a "Start/Stop" button, which will show "Start" initially because the computer has already activated the SPGAS software. Touching the button alternately "starts" and "stops" the operation of the spectrometer.

At the lower right position are two displays: the upper is labeled "Show Spectra" with options for "Background," "Sample," and "Absorbance." Below it is a display labeled "Collecting:" which indicates one of the following operations: "Background," "Moving Mirrors," or "Sample." Below it is a time line showing the "Time Left" for each of those three operations. The SPGAS software is causing the FTIR and Transfer Mirrors to cycle repeatedly through those three

Figure 4. Start-Up Screen Display



operations. With each completed cycle, the SPGAS performs a quantitative analysis of the collected data to generate the screen displays of spectra.

At the “Show Spectra” display, one chooses which of the three options one wants displayed on the main portion of the screen. One can switch back and forth between the current Background spectrum, the current Sample spectrum, or the calculated Absorbance record.

At the top right of the screen, just below the “SpectraStream” logo is a scan bar, which shows on a 0 to 100% scale the percentage of the selected number of scans that have been performed by the FTIR.

#### 4.5 SPGAS/SpectraStream Software Displays

A typical SPGAS screen display is shown in Figure 5. It provides a graphical display of the concentrations of the selected gaseous species as a function of time. The gaseous species are those prescribed in advance to the factory for the inclusion of HITRAN calibration data. For example, Figure 5 shows plots for CH<sub>4</sub>, CO, CO<sub>2</sub>, and H<sub>2</sub>O.

Also presented at the bottom of the screen is the “Concentration Log,” for the selected molecules. Two sets of concentration and standard error numbers are listed. The first set to the left are the concentration in ppb and its associated standard error from the long-term or multiple scan collection of data. The second set to the right presents the “FCT” (fast concentration tracker) results and associated standard error. The FCT numbers are generated by SpectraStream from single scan data collection to provide the fast-response, early-warning detection of changes in gas composition or impurity levels. The associated standard errors are greater because fewer data points are used in the calculations. These numbers are continuously updated as the SPGAS analyses proceed.

Above the graphical display are one or more buttons for the selected calibration gases. Touching one of these buttons brings up a display for that gas of both the multiple scan and the fast scan concentrations as a function of time. One such display is shown in Figure 6. The solid curve presents the multiple scan results and the dashed curve presents the single scan results.

#### 4.6 System Test

There is one easy system test that will reveal if the IRGAS-XSA is performing correctly. It is based upon displays of single beam background data curves. This test determines if the nitrogen purging of the spectrometer and optics has reduced the background moisture level to a minimum level.

To perform this test, display again the “Background” single beam record and look at the Background Moisture Index (BMI) located at the lower left corner of the plot. If the magnitude of the BMI is less than  $5 \times 10^{-3}$ , adequate purging has occurred and one can proceed to sampling process gas for analysis; if not, then more nitrogen purging may be required.

Figure 5. Typical Screen Display of Concentration vs. Time

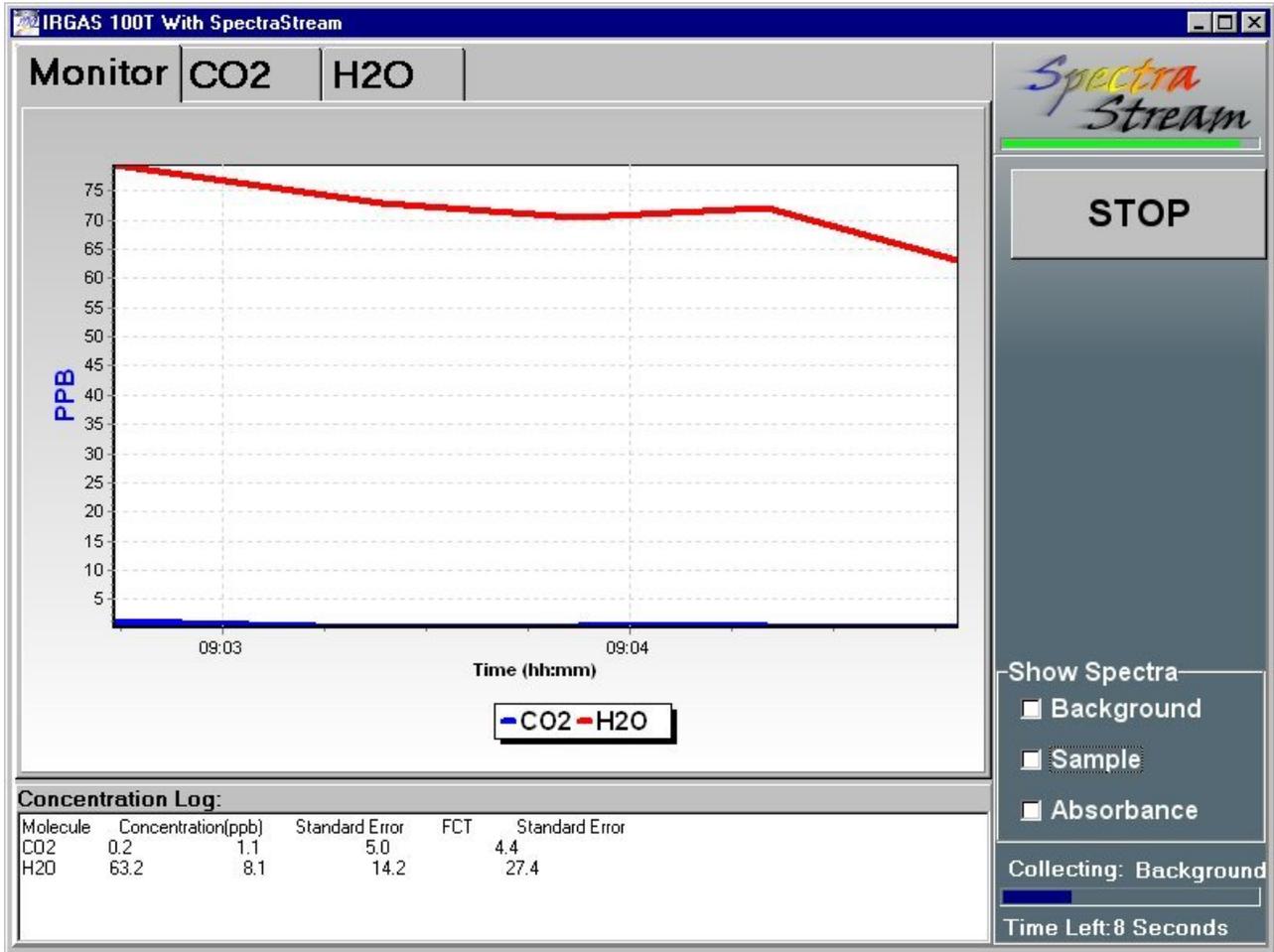


Figure 6. Multiple and Fast Scan Concentration Displays

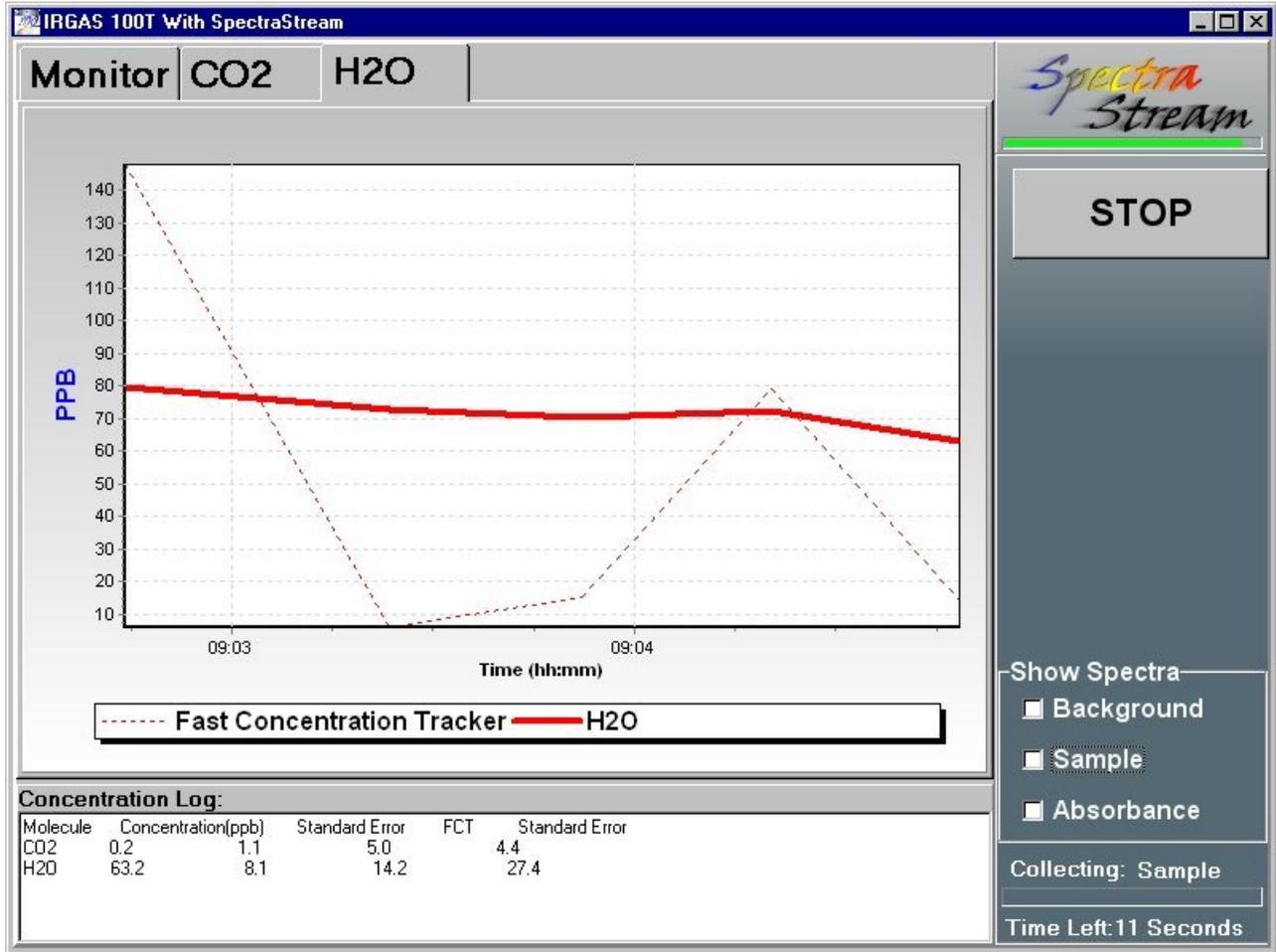


Figure 7. Single Beam Background Record

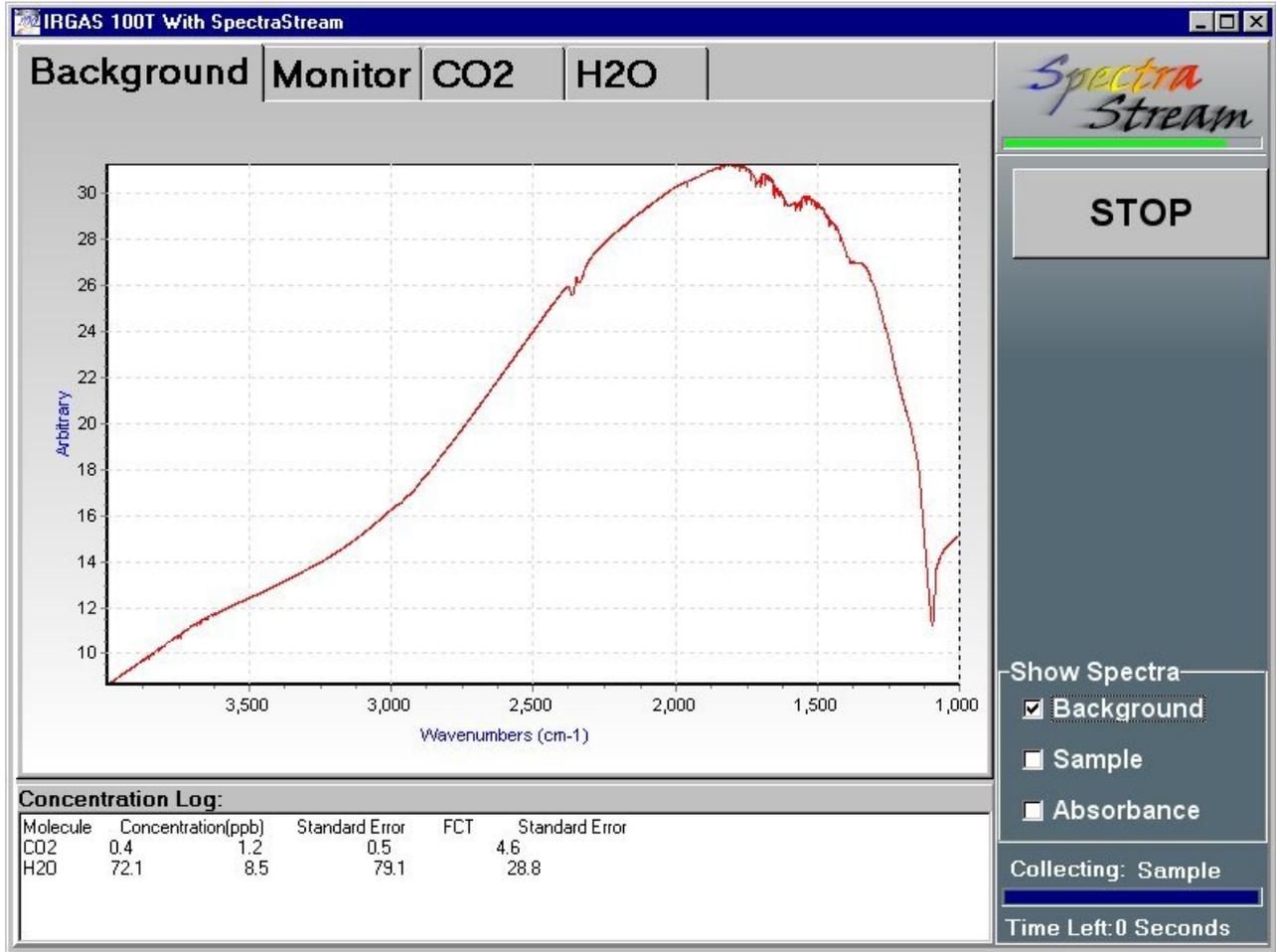
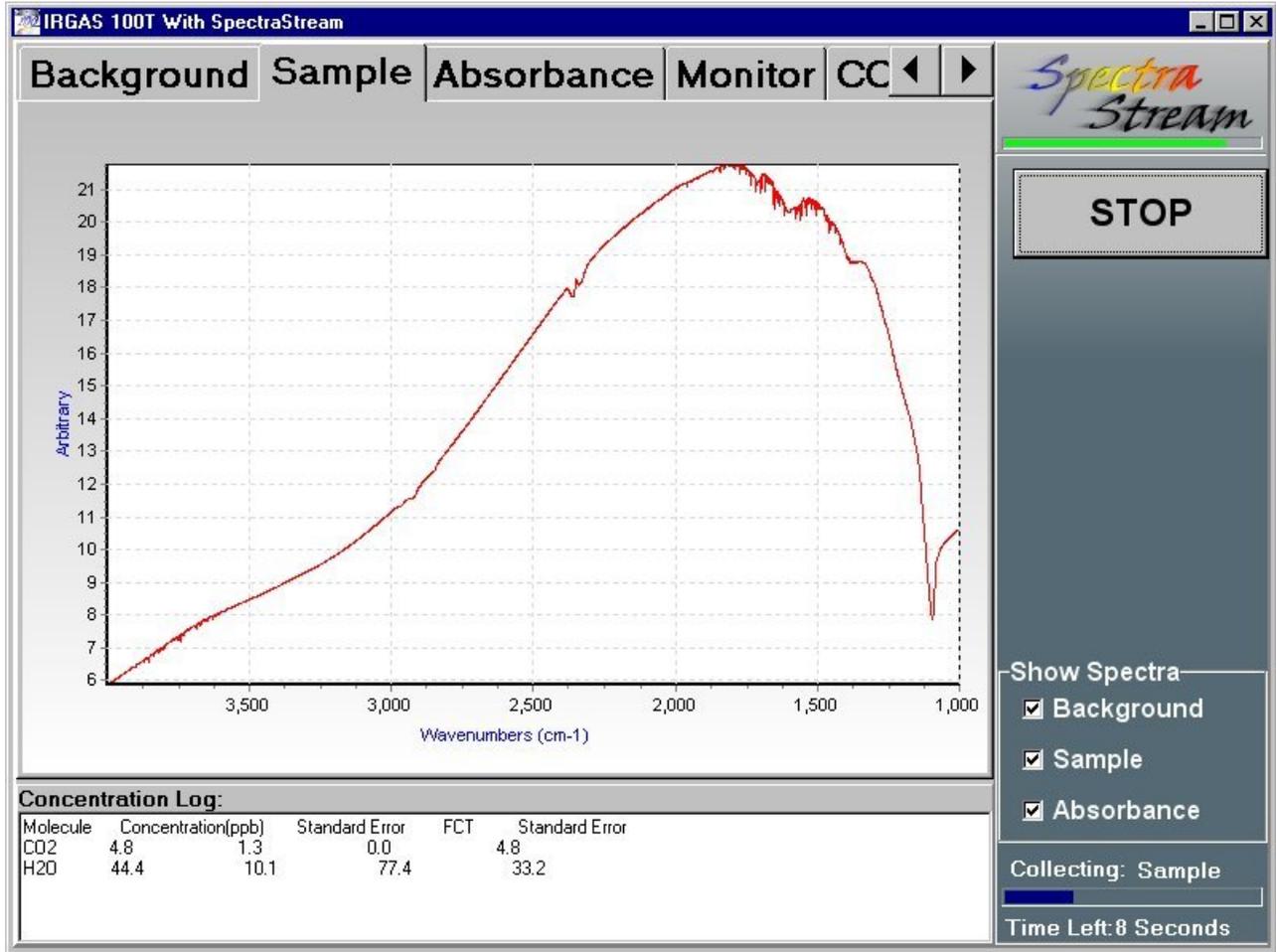


Figure 8. Single Beam Sample Record



## 5.0 Full System Operational Procedures

### 5.1 Gas Cell Purge

The gas cell should have been purged with ultra-pure nitrogen prior to and between the additions of the sample gases.

### 5.2 Automated Data Collection and Display

As indicated in the Start-up Procedures, once the System is turned on, data collection begins immediately and continues to cycle between Background Data Collection and Sample Data Collection with a Quantification Analysis of Absorbance performed for each complete cycle. The Absorbance data are then converted to concentrations via the HITRAN gas calibrations and the resulting concentration values are displayed on the screen as a function of absolute time.

The IRGAS-XSA is based upon the System Operator being an active observer of the displays of gas concentrations to determine if departures from expected concentration levels occur. Such departures may occur due to changes in the quality of the supply gases, deterioration of inline gas purifiers, leaks in the gas lines or gas manifold components, degradation of seals in the process tool, alterations in the operating performance of the process tool, etc.

The IRGAS-XSA is also dependent on the System Operator keeping an independent log of operational changes with the process tool as a function of absolute time; this is necessary so that data archived by the IRGAS-XSA can be correlated at any later time with the performance of the process tool.

### 5.3 Recording, Archiving and Recalling of Collected Data

Each cycle of Background, Sample, and Absorbance Data is automatically stored in a Grams SPC file format in archive folders on the hard disk of the CPU. The spectral records are stored in archive folders (D:\Irgas\Spectral Records) as the spectra are collected.

#### “Spectral Records” Filename Convention:

Background\_”TimeStamp”.spc  
Sample\_”TimeStamp”.spc  
Absorbance\_”TimeStamp”.spc

“TimeStamp” = “YY-MM-DD HH-MM-SS”

A file is created about every 30 seconds.

The “Spectra” files are stored inside a zip file in ½-hour increments to keep the file size manageable at < 1.4 mb:

BackgroundTimeStamp.zip  
Sample.TimeStamp.zip  
Absorbance.TimeStamp.zip

The “zip” files in turn are stored inside a folder with the creation date as their name:

D:\IRGAS\Spectral Records\”Date”\ where “Date” = “YY-MM-DD”

“Quantification Log” Filename Convention:

Quan\_”DateStamp”.Log

“DateStamp” = “YY-MM-DD”

The Quantification Log files are text files that are easily readable by software like MS Excel. A quantification is produced every 30 seconds and stored inside its Quantification Log file. One Quantification Log file is produced per day. These files are stored inside a folder with the year and month as their name:

D:\IRGAS\Quantification Log\”YY-MM”\

The quantification calculations are stored in separate archive folders (D:\IRGAS\Quantification Log) in a similar manner. Files are retrieved by their date and hour identification. The file sizes have been defined for content size corresponding to a floppy disk, so that individual folders can be downloaded.

#### 5.4 Downloading Archived Data Records

It becomes necessary for the System Operator to download archived data about every 30 days in order to free-up space on the hard disk. Use MS Windows standard procedures to copy files to floppy disks. A more efficient way is via the Ethernet 10/100 port for transfer of the data to the System Operator’s in-house network; the latter likely requires engaging the assistance of the internal IT Department.

## **6.0 System Shutdown Procedures**

### **6.1 Temporary Shutdown**

If the IRGAS-XSA is being shut down only temporarily and will be used again in the near term, then the System switch should be left on so that the FTIR spectrometer power remains on to keep the internal temperature above ambient. The Computer and Heater switches may be turned off. It is also desirable to keep the nitrogen purge gas flowing through the spectrometer, transfer optics, and separately through the gas cell.

### **6.2 Long Term Shutdown**

Before turning off all power switches and terminating flow of purge nitrogen, the gas cell should be purged thoroughly with nitrogen using a purge, pump-out, bakeout procedure to rid the gas cell and process gas lines of all traces of the process gas; three cycles of purging and pumping with the gas cell temperature elevated is probably adequate. Allow the cell to cool to ambient temperature with purge nitrogen before stopping the flow and then cap the input and output lines with VCR caps and seals.

### **6.3 Moisture Protection**

The above procedures are designed partly to remove all adsorbed moisture from the gas lines and gas cell. Residual moisture in the presence of acid gases can be very destructive to the mirrors in the gas cell.

## **7.0 Packing for Shipment**

The IRGAS-XSA should be repacked for shipping forward in the same manner as it was received. That entails placing it back on the custom pallet so that the wheels are protected, replacing the top cardboard cover, tying it down with the four nylon straps, and reinstalling the panels of the wooden crate.

## **8.0 System Diagnostics and Technical Support**

### **8.1 User Tests**

Repeat the tests described in Section 4.6 to ascertain if the System is performing correctly; call for Technical Support if there are any major departures from the prescribed results.

There is a built-in diagnostic to determine if the transfer mirrors in the gas cell purge box are functioning properly—that is, providing the movement in and out of the IR beam position. This diagnostic is provided at the following location: D:\IRGAS\PurgeBoxDiagnostic.exe

### **8.2 Telephone and Email Support**

Telephone technical support is available Monday through Friday, 8:30 am to 5:30 pm U.S. Mountain Time by calling 505-343-1489, 343-9520, or 343-9500; fax 509-479-2980.; SKYPE: cicp-jorge

Inquiries may also be sent at anytime to: jperez@cicp.com with copy to ebonano@cicp.com.

### 8.3 Trouble-shooting

#### 8.3.1 Gas Cell Degradation: Mirrors, Seals, Windows

The best way to track any degradation of the gas cell mirrors and windows is to keep a daily or weekly record of the energy throughput in a ratio measurement of the single beam magnitude for the IR beam through the empty cell (a “Sample” reading) compared to that for the IR beam bypassing the cell (a “Background” reading). When that ratio decreases to less than two-thirds of the value recorded for the cell when brand new, then there is some indication that either the windows are becoming coated or the mirror reflectivity is diminishing. At that point, fax or email your records to CIC Photonics for examination.

Occasional testing of the gas cell and plumbing lines with a helium leak detector will provide assurances that the VCR fittings, C-seals, and Kalrez O-rings are intact. Long term exposure of the Kalrez O-rings to high temperatures and acid gases can lead to their degradation.

#### 8.3.2 Temperature Controller and Heater

If there is indication that the gas cell is not being heated, first check the fuse on the temperature controller; a blown fuse may indicate a short in the wiring to the heater. Using an ohm-meter, then check the resistance across the leads to the heater; a good heater should read approximately 50 ohms.

Do not overdrive the heating element by exceeding the temperature limit; the silicone rubber composition of the sheet heater will start to degrade at 300°C.

#### 8.3.3 FTIR Spectrometer

**For proper operation, the spectrometer operating temperature must be kept at or below 30°C and should not exceed 40°C.**

#### 8.3.4 CPU and Monitor

The IRGAS/SPGAS software is based upon a standard Windows NT/2000/XP Operating System; any IT Department should be able to trouble shoot it if necessary; or call CICP Technical Support.

To shut down the CPU, first touch the “STOP” button on the screen; then click “OK” at the screen message and turn off the Computer switch.

If using a touchscreen monitor, and the touchscreen appears not to be functioning, look at the Mouse symbol in the lower right bar on the screen; observe whether the RED indicator is on the left side of the mouse; if not touch the top left of the Mouse to activate it. The Mouse symbol operates like a physical mouse with left and right buttons, which you can toggle back and forth.

To use an on-screen keyboard, touch the Keyboard symbol on the lower right bar.

### 8.3.5 Nitrogen Purifier and Gas Lines

The expected lifetime of the nitrogen moisture purifier under normal continuous operation of the system is six to nine months, so long as it is not exposed to air. Indication that it may be becoming saturated with moisture comes from a steady increase in the measured moisture content in the “Background” records. When this occurs to the extent that the background moisture is significantly interfering with quantitative measurements of moisture as an impurity in the process gases, the nitrogen purifier should be replaced.

### 8.3.6 Electrical System: Fuses,

The system electrical circuitry is protected with a 3 amp fuse. The FTIR spectrometer is fused independently. If any of these fail, a thorough check of the associated circuit should be conducted.

### 8.3.7 Vibration Isolators

The FTIR spectrometer sits within a small mounting frame containing handles, for easier lifting, and vibration isolators, for damping out room vibrations.

## 8.4 Removal of Gas Cell for Warranty Repair

Should it become necessary to return the gas cell due to degradation of mirrors, windows, or seals, please call Technical Support for instructions. The gas cell can be removed and returned without having to return the entire IRGAS system. In brief what is involved is (1) flushing the cell well with purge nitrogen, (2) removing the black anodized cover box which encloses the gas cell, (3) disconnecting the VCR fittings and capping them, and (4) lifting the cell off the spectrometer and out of the system enclosure. Then special packing and shipping procedures are to be followed.