



Photronics

**Model 400 Series Spectrophotometer
User's Manual**

P/N 1656

S.I. Photonics, Inc.

Tucson, Arizona

Table of Contents

1. Safety Message.....	1
2. Introduction.....	3
2.1 Items Included in 400 Series Systems.....	4
3. Technical Assistance, Service, and Spare Parts	5
4. Quick Installation Instructions	7
5. Initial Setup of the 400 Series Spectrophotometer.....	9
5.1 Removing the Cover	9
5.2 Inside the Spectrophotometer	10
5.3 Initial Inspection.....	10
5.4 AC Voltage Selection.....	10
5.5 Installation of Probe Holder	10
5.6 Installation of Fiber Optic Sampling Accessories	10
5.6.1 Fiber Optic Probe Installation	11
5.7 RS-232 Communications Link	11
5.7.1 Configuring the Communications Link	11
5.7.2 RS-232 Cable	12
5.7.3 Baud Rate Selection.....	12
5.8 Spectrograph Warm-up	13

6. Software Installation	15
6.1 Host computer Hardware and Software Requirements	15
6.2 Installing the SI 400 Application	15
6.2.1 Windows 9x, 2000, XP	16
7.0 Application Software	17
7.1 Starting the SI 400 Application	17
7.2 Graphical Displays and Associated Controls	18
7.3 On-Line Help	20
7.4 Function Keys	20
7.5 Setting Baud Rate and COM Port for Host Computer	21
7.6 Initializing the Spectrophotometer	22
8. Sample Interface Options	23
8.1 Fiber Optic Dip Probe	23
8.1.1 Using Standard Dip Probe	23
8.2 Acquiring Absorption Spectra	24
8.3 Cuvette Cell Holder	24
8.4 Fiber Optic Reflectance Probe	24
8.5 External Shutter and Lamp Connectors	25
8.5.1 External Shutter Connector	25
8.5.2 External Lamp Connector	26

8.6 Integrating Sphere Accessory	26
8.6.1 Attaching Integrating Sphere	26
8.6.2 Integrating Sphere Operations	26
9 The SI 400 Main Front Panel	29
9.1 Graphical Display	29
9.2 Main Front Panel Controls	30
10 Quantitation	39
10.1 Standards and Unknowns Tables	40
10.2 Graphical Display	40
10.3 Quantitative Analysis Procedures	40
10.4 Quantitative Front Panel Controls	42
11 Timed Acquisition.....	45
11.1 Timed Acquisition Front Panel Controls	46
12 Peak Find.....	51
12.1 Graphical Display	51
12.2 Peak Find Panel Controls	52
13 Multi Spectra Display	53
13.1 Graphical Display	54
13.2 Multi Spectra Display Front Panel Controls	54

14 Lamp Adjustments	57
14.1 Adjusting Lamp Intensities	57
14.1.1 Deuterium Lamp Intensity	58
14.1.2. Tungsten Lamp Intensity	59
14.2 Tungsten Lamp Replacement	59
14.2.1 Adjusting Tungsten Lamp Current	60
14.3 Deuterium Lamp Replacement	61
14.4 Lamp Warm-up Times	62
15 Specifications	63
16 Trouble Shooting	65
16.1 Error Messages	65
16.2 Unreliable Communications	66
16.3 Screen Size	67
16.4 Lamp Intensity Warning Messages	67
16.4.1 High lamp Intensity	68
16.4.2 Low Lamp Intensity	68
17 Index	69

1. Safety Message

CAUTION!

It is necessary to remove the cover of the 400 Series Spectrophotometer for some procedures described in this manual. In some cases, power to the instrument must be applied while adjustments are made. There are two regions of the instrument power supplies that are shielded and marked where dangerous voltages exist. Avoid touching any components behind these shields. If the shields are removed for any reason, re-install them before power is applied to the instrument.

2. Introduction

The S.I. Photonics 400 Series Spectrophotometers are laboratory instruments that utilize the latest solid state CCD array detector technology. The optical system is specifically designed to be used with fiber optic sampling accessories. A variety of probes and probe configurations are available for different sample requirements.

The instrument consists of tungsten and deuterium (on model 420 and 440 only) light sources, optical shutters for the light sources, a spectrograph with integrated CCD array detector, and the detector control electronics.

The tungsten source is part of the main power supply printed circuit board. The lamp is a miniature quartz-halogen type. The lamp output is optically regulated using a photodiode detector and feedback circuit. The lamp power supply also features over-current protection during both lamp start-up and operation. The tungsten lamp is used for the 350 to 980 nm range when the deuterium lamp is not used, and from 380-460 to 980 nm when the deuterium lamp is used.

The deuterium lamp is a high output and long life unit capable of 4000 hours of operation. The lamp bulb is pre-aligned to a flange, which makes changing the lamp a simple operation. The lamp power supply on and off functions are performed under software control so that the lamp is turned on only when the user needs to acquire data in the 190 to 460 nm range.

The spectrograph is a sealed all-reflective optics system, using a single mirror for collimating and focusing, and a 325-line/mm diffraction grating blazed at 300 nm. An integrated, fixed order-sorting filter is used to eliminate second order radiation. The spectrograph optics, CCD array detector, and detector electronics are enclosed in an insulated, temperature regulated enclosure to ensure high baseline stability.

A digital signal processor is used in the 400 series instrument to execute the commands issued by the host computer. This very high speed 24 bit processor directly generates the timing states necessary for CCD readout, sums the data from multiple CCD reads, and controls other devices in the spectrophotometer such as optical shutters and deuterium lamp power relays. The processor has an integrated serial communications interface that is used to transfer command, status, and data information between the spectrophotometer and the host computer.

This User's Manual covers the installation, use, and maintenance of the S.I. Photonics 400 Series Spectrophotometers. It is valid for models 410, 430 (tungsten lamp source only) and 420, 440 (tungsten and deuterium lamp sources).

2.1 Items Included in 400 Series Systems

Basic items shipped with your instrument are listed below, see the packing slip for a detailed list.

- 1) AC power cord
- 2) 6' long DB9M to DB9F RS-232 cable
- 3) DB9M to DB25F adapter
- 4) Fiber optic sampling accessory
- 5) User's manual
- 6) Software installation CD-ROM
- 7) Spectrophotometer
- 8) Spare tungsten lamp
- 9) Trimpot adjustment tool
- 10) Hex wrenches (2)

Verify the contents of the shipping container and notify S.I. Photonics if any items are missing. We can be reached at (520)293-6911 or via the internet at **www.si-photonics.com**

3. Technical Assistance, Service, and Spare Parts

If it is necessary to contact S.I. Photonics for support, please have ready the instrument model number, serial number, or optics serial number and control software revision number. Print out the **About Spectrometer** window from the **Help** menu for the information. The model and serial numbers can also be located on labels on the inside of the instrument base. The software revision number is printed on the distribution CD label and can also be found in the View menu of the main front panel under **VI info**.

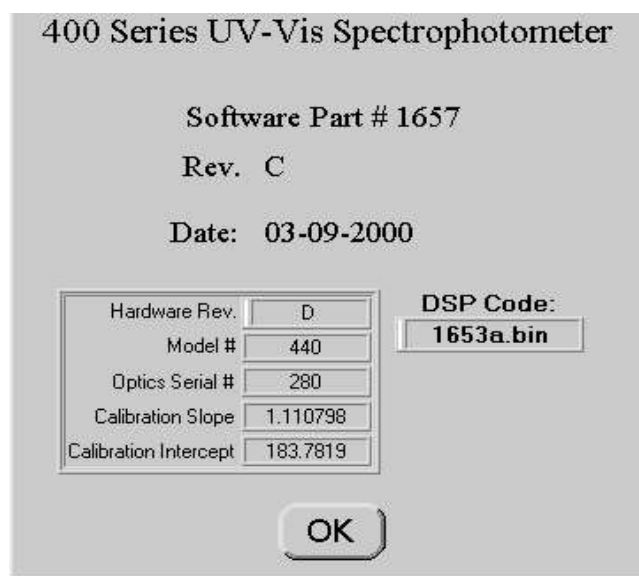


Figure 3-1 About Spectrophotometer Window

Replacement parts for the 400 Series Spectrophotometers are:

Part Number	Description
1991	Lamp, Tungsten, Quartz
1103	Lamp, Deuterium

The tungsten lamp is part number Thorn M29 (product no. 72170T) distributed by Microlamp, Ft. Lauderdale, Florida and may also be purchased directly from them. It is recommended to have a spare tungsten lamp.

For technical support and to order supplies and replacement parts contact the Service Department at (520) 293-6911 or via email at support@si-photonics.com.

4. Quick Installation Instructions

This section is for those who either enjoy the challenge of setting up a new instrument without reading the manual, or do not have the patience to read a manual thoroughly before plunging in. If you are this type of person, and you have experience installing scientific instruments and Windows software, read this section to get up and running quickly. Then, when the excitement has worn off or you get stuck, go back and thoroughly read the manual. For those persons that are more cautious, read the manual Sections 5 through 8, then follow the instructions to get the instrument operational before continuing with the detailed software description.

Before you plug in AC power to the instrument, remove the cover by loosening (not removing) the four screws on the ends. If you are using 220VAC power, the power selection switch (located near the main power switch on the main power supply board) must be switched and the fuse value changed.

Install the probe holder by sliding it onto the probe stand. If equipped with a probe, install the fiber optic probe next. Make sure that the ends of the fiber connectors are clean (wipe with alcohol and lens paper), and that the SMA connectors are plugged into the right places. A nylon-tipped setscrew is used to hold the connector ferrules in the sleeves. Tighten the setscrews using the supplied hex (Allen) wrench. Replace the cover on the spectrometer and attach the AC power cord. Connect the RS-232 port on the spectrometer to an unused port on the host computer.

Install the software in windows. When the CD is loaded the program install should automatically start. If the install does not start automatically, execute the **SETUP.EXE** on the CD-ROM and follow the directions. If you are unsure about what hardware you need to run the application, refer to Section 0.

Once installation is complete, start the **SI400** application, and turn on the spectrophotometer power. The tungsten lamp should immediately light, but the deuterium lamp on model 440 instruments will not light until commanded by software.

Click the OK box to initialize the instrument. If the instrument is not communicating with the host computer, an error message will pop up. To correct error pull down the **Operate** menu from the main screen and click on the **Configure** option. Refer to section 14 for instruction to adjust light intensity errors.

Select the COM port that you are using on the computer. Leave the baud rate set to 38,400, as the spectrometer is shipped with this baud rate. Choose printer

type, Monochrome or Color. Click the **Save** button to keep any changes, and then select from the **Operate** menu the **Initialize** item.

At this point, if everything has gone just right and the computer and spectrophotometer are communicating, you should hear a faint click-clack from the tungsten lamp shutter. Also, the **Blank** button in the upper right corner of the screen will change from a light gray inactive mode to a red active mode. The Setup file is automatically read from the hard drive and the operating parameters of the spectrometer will be set. The parameters include the wavelength range and the lamp crossover wavelength. The wavelength scale on the main spectrum display will reflect the wavelength range. At this point, the wavelength range and lamp crossover wavelength can be changed if desired by selecting the **Wavelength Range** item, which is located in the Operate menu. If you have a model 440 instrument and choose a minimum wavelength below the lamp crossover wavelength, (460 nm is the initial default), you will be asked if you want to turn on the deuterium lamp.

If you get error messages instead at this point, follow the advice of the error messages. If this does not help, read Section 16 of the manual.

Once the spectrophotometer is operating, immerse the probe in water, and start pushing buttons. The **Blank** takes a blank spectrum, and **Sample** takes a sample spectrum and records the results on the screen. Most of the buttons are self-explanatory in the usual Windows fashion. The **Quantitative** Item in the Operate menu, brings up a new screen for quantitative analysis. The **Timed Acquisition** item in the Operate menu opens a new screen where entire spectra or discrete wavelengths can be acquired at set time intervals. The **Multi Spectra Display** item can be accessed from the **View Menu** and allowing up to 10 spectra to be viewed and manipulated.

When you are ready for more detail read the manual Sections 7 through the end. This way you will be able to use the software to its fullest capabilities, and change lamps and adjust lamp intensity when the need arises.

5. Setup of the 400 Series Spectrophotometer

The 400 Series Spectrophotometer includes a fiber optic probe that is not connected during shipment. It is necessary to connect the probe in order to conduct the initial system tests. The probe stand and sliding probe holder is designed to conveniently hold the probe when it is not in use, or when sampling the contents of small containers. It is not essential for the operation of the spectrophotometer and may be removed if desired.

5.1 Removing the Cover

The spectrophotometer cover must be removed to install the probe. Before starting, make sure that the AC power cable is disconnected where it enters the instrument. Next, locate the four cover screws, two on each end of the instrument. Using a Phillips screwdriver, loosen the four screws approximately 5 turns, or 1/8". The screws should not be removed. Next, lift the cover **straight up** until it clears the internal components, and set it aside.

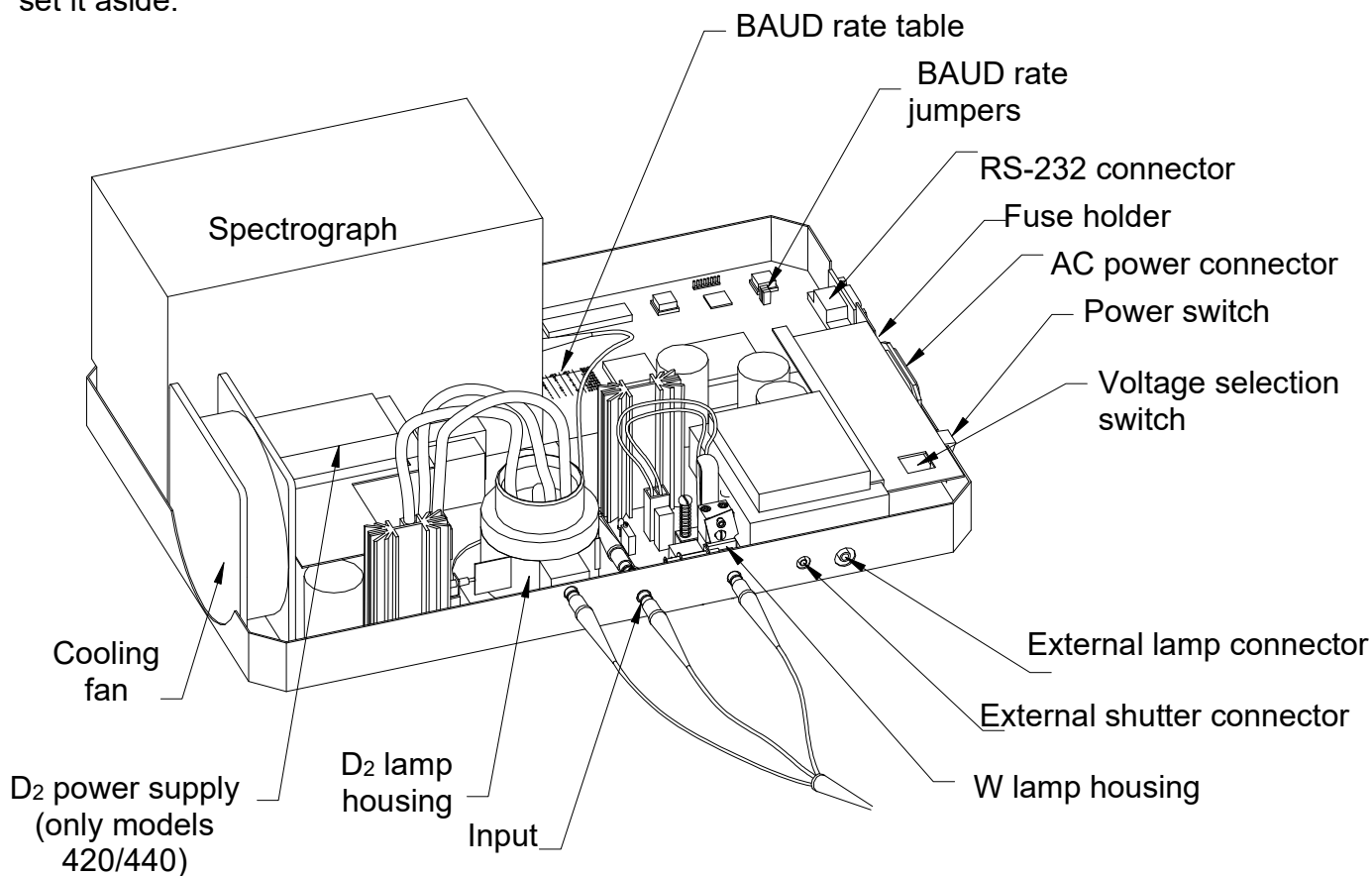


Figure 5-1 Main compartment of the 440 Series Spectrophotometer

5.2 Inside the Spectrophotometer

Inside the spectrophotometer is a compact spectrograph (which is thermally insulated for temperature stability), a digital electronic controller card, and the main and optional deuterium lamp power supplies as shown in Figure 5.1.

5.2 Initial Inspection

With the cover removed, inspect the inside of the spectrophotometer for any parts that may have vibrated loose during transit.

5.3 AC Voltage Selection

The spectrophotometer is configured to operate from 100 to 120VAC mains when delivered from the factory. If the instrument is to be operated from 220VAC mains, the AC voltage selection switch must be changed. This switch is located beneath a guard near the power switch. The switch is accessible through a hole in the guard. A small flat blade screwdriver is used to change the switch setting.

5.4 Installation of Probe Holder

To install the probe holder on the probe stand rod, squeeze the flat metal spring stop slightly and slide it first over the rod end. The black plastic surface should now be on top.

5.5 Installation of Fiber Optic Sampling Accessories

All sampling accessories for the 400 Series Spectrophotometer are fiber optically coupled to the instrument. Each accessory will have either two (UV only or Visible only) or three (UV/Visible) fiber optic connectors. The standard probe supplied with the 400 series instrument is a fiber optic dip probe. Also available with the instrument are the Universal Fiber Optic Sampling Bench, which can be fitted with a cuvette holder or optical filter holder, and a fiber optic reflectance probe.

The 400 series instruments are designed to be used with probes terminated with standard SMA type connectors, such as the standard fixed path length dip probes. The connectors are not attached to the instrument in the conventional manner of tightening the retaining nut, but rather the connector ferrule is clamped in place using a nylon-tipped setscrew. This approach was taken to ensure a more mechanically stable connection. It is important to use only the supplied nylon tipped setscrews to clamp the connector ferrules. Standard setscrews will damage the ferrule, and possibly break the fiber inside the connector.

Fiber optic probes are fragile in some operations. Use caution with the probe as damaged probes can not be easily repaired; replacement is usually the only alternative. The fiber optics in the probe are fragile and can be broken if mishandled by placing too great of tensile stress, by exerting too small of bend radius on the cable sheath, or by kinking or crushing the sheath. Never swing the probe from the fiber optic sheath, or pull on the probe when attached to the spectrophotometer such that the sheath is stretched tight.

5.6 Fiber Optic Probe Installation

The fiber optics at the connectors must be clean, free of dust and especially fingerprints (to avoid UV absorption) before they are installed. Use lens tissue moistened with isopropanol to clean if necessary.

To install any of the sampling accessories, locate the small hex (Allen) wrenches provided with the instrument. Remove the plugs in the spectrometer where the fibers attach, and remove the caps from the ends of the fiber optic ferrules. Take care not to scratch or damage the ends of the fiber optics when the protective caps are removed. Insert the W source fiber into the W source bushing until it stops and secure it with the setscrew. Install the Input fiber (and for model 420/440 instruments, the D₂ source fiber) in the same manner.

5.7 RS-232 Communications Link

The communications channel between the host computer and the 400 Series Spectrophotometer is an RS-232 serial link. Information passed over this link includes commands to operate light shutters, initiate the CCD array detector readout, turn on or off the optional deuterium lamp, etc. The link is also used to pass the light intensity as a function of wavelength information used to determine the optical transmission or absorbance of a sample. One important task for the communications link is to initialize the spectrophotometer by passing a software program to its microprocessor. This task must be accomplished before the spectrophotometer can recognize any commands or perform any functions.

5.7.1 Configuring the Communications Link

In order for the communications link to pass information, it must be properly configured involving both the physical cabling of the host computer COM port to the spectrophotometer, and ensuring that the two pieces of equipment are communicating at the same data rate, or baud rate. All other communications parameters, such as number of data bits, parity bits, etc. are permanently configured in the spectrophotometer, and are configured in the host computer by the SI400 application without the need for user intervention.

5.7.2 RS-232 Cable

The communications cable must be a high-quality, shielded cable intended for RS-232 use, such as the one supplied with the instrument. This is especially critical when higher baud rates (19,200 and higher) are used. The cable supplied with the 400 series instruments is a 6' DB9 male to DB9 female, wired straight through (pin 1 to pin 1, pin 2 to pin 2, etc.) which can be connected to a DB9 male COM port connector on the host computer. A DB25 female to DB9 male adapter is also supplied to allow connection to a DB25 male serial port on the host computer. It is possible to use a longer cable, however it may be necessary to use a lower baud rate to ensure reliable communications.

Connect one end of the cable to the spectrophotometer and the other to any unused COM port on the host computer configured as COM1, COM2, COM3, or COM4. If all COM ports on the computer are in use, it may be necessary to add a COM port connector or interface card. It is important that the COM port to be used is not sharing an interrupt. Refer to your computer equipment supplier for information.

5.7.3 Baud Rate Selection

It is advantageous to use as high a baud rate for the communications link that reliably works as spectral data will be transferred from the spectrophotometer and be displayed or analyzed faster. Under some conditions it may be necessary to decrease the baud rate to ensure reliable communications with some host computers.

The COM ports for most modern PC compatible computers are capable of reliable operation at baud rates of at least 19,200 if the cable length is kept short. If it is necessary to use a cable of approximately 15' or greater between the host computer and the spectrophotometer, or if an older PC computer that can not support 19,200 baud is used, it will be necessary to decrease the baud rate to 9,600. If the host computer is capable of communicating at 38,400 baud or higher, and only the 6' cable supplied with the instrument is used, it may be advantageous to increase the baud rate.

The baud rate for the 400 Series Spectrophotometers is set to 38,400 at the factory. With a 16550 UART serial port, baud rates up to 57,600 are possible

To change the baud rate, it is necessary to remove the cover of the spectrophotometer (Refer to Section 5.1 Removing the Cover) and change the baud rate jumpers. The jumpers are on the controller board near the RS-232 connector. There are 3 pairs of pins emerging from a small block labeled JP1, JP2, and JP3. A plastic plug containing a wire fits down over a pair of pins electrically connecting them (Closed or 1) or is removed or pushed down over only one pin (Open or 0). Select the desired baud rate by positioning the

jumpers as shown in the table 5-1 below. This table is also printed on the controller board near the spectrograph.

JP3	JP2	JP1	BAUD Rate
0	0	0	9600
0	0	1	19200
0	1	0	38400
0	1	1	57600
1	0	0	115200
1	0	1	230400
1	1	0	460800
1	1	1	921600

Table 5-1 Baud Rate Jumper Table

The image shows a software configuration window with the following elements:

- Serial Port:** A pull-down menu currently set to "COM 2".
- Baud Rate (Std):** A pull-down menu currently set to "38400".
- Printer Type:** A button labeled "Monochrome".
- Adjust D2 Lamp:** A button.
- Serial Port Type:** A pull-down menu currently set to "Standard".
- Save:** A button.
- Close:** A button.
- Adjust W Lamp:** A button.

Table 4-2 Configuration Pull Down Menu

5.8 Spectrograph Warm-up

At this point, the spectrophotometer cover is ready to be replaced. Follow the cover removal procedure in reverse order, then connect the AC power cord supplied to the instrument and to a standard 110VAC wall outlet.

The spectrograph in the 400 series instruments is temperature regulated at approximately 35°C to improve baseline stability. A warm-up time of approximately 30 minutes is required for the temperature of all optical elements to equilibrate. During this time, the baseline will drift. If careful measurements are to be made, or if long periods between blank and sample acquisition are anticipated, temperature equilibration and warm-up is essential.

6. Software Installation

The host computer using the SI400 series application software controls the 400 Series Spectrophotometer. This software system operates in the Windows 95 or later environment and conforms to normal Windows operating conventions whenever they apply. Users unfamiliar with the Windows environment should refer to the "Getting Started with Microsoft Windows" and "Microsoft Windows User's Guide" that is part of the Windows distribution package before proceeding.

6.1 Host Computer Hardware and Software Requirements

The SI400 control application runs under Windows 95, 98, NT, 2000 and XP operating systems. Minimum hardware requirements include:

- 1) a Pentium 90 CPU
- 2) a hard disk drive with a minimum of 6 MB free space
- 3) a CD-ROM Drive or 1.44 MB 3.5" floppy drive
- 4) a two-button mouse (or a three button mouse in two-button mode)
- 5) a minimum of 32 MB RAM
- 6) one RS-232 serial port equipped with a 16550 buffered UART configured as COM1, COM2, COM3, or COM4 using the standard Windows addresses and interrupt vectors. The port can have either a DB9 or DB25 male connector.

****Alternatively, an RS-232 to USB conversion cable is available for PC configurations utilizing USB input.**

A VGA monitor with a minimum 800 X 600 resolution is required.

Throughout this manual, a basic knowledge of the Windows environment and its conventions is assumed. If necessary, refer to the Windows User's Manual for guidance.

6.2 Installing the SI400 Control Software

The SI400 Control Software is distributed in compressed form on a CD-ROM. The files must be installed onto your hard disk drive using and Setup program on the CD-ROM from Windows. *Disks can be made from the CD-ROM – Call S.I. Photonics for instructions.*

6.3 Windows 9X, NT, 2000 and XP

To install, insert the CD-ROM into your drive.

The install set-up window should open automatically, if not, go to the **Start** Menu and click on **Run**. Type in CD-ROM drive designation (i.e. D:) A window will appear Click on SET UP.

Press the Install button and follow the prompts to complete the installation.

7. The SI400 Control Software

The SI400 software application uses the *Virtual Instrument* (VI) concept where the various buttons, knobs, dials, displays, and controls that you would expect to find on an instrument along with pull down menus have been replaced by their graphical equivalents that appear on the computer screen. The instrument hardware no longer has physical controls, but the combination of computer screen, keyboard, and mouse have replaced their function in ways that can be easily optimized for specific tasks. Different front panels can exist to perform different functions for the same physical hardware! The SI400 application has different front panels for general data acquisition, quantitative data acquisition, and for acquiring data at fixed time intervals, each optimized for the task.

7.1 Starting the SI400 Application

During installation a program group was created and can be accessed in Windows 95/98/NT/2000 and XP from the Programs item on the Start menu. To start the application, SI 400 program group icon, then double click on the SI400 application icon. If you do not select to have a program group created, execute the SI400.exe program using the file manager.

When the SI400 application is started, it presents the main VI front panel that appears (see Figure 6-1 Main front panel) to have an assortment of buttons and other digital controls and drop down menus, along with a graphical display of optical absorbance , transmission or reflectance. The buttons are "pressed" by using the mouse to point to the button, and pressing the left mouse button. If a button is active, it will react when pressed so that it looks like it was pressed, and that some activity is being performed.

There are times when a button cannot become active until some other function is first performed. For example, the **Blank** button is inactive until the instrument is initialized. The **Sample** button is inactive until a blank has been acquired. When a button is inactive, it is a light gray, semitransparent color.

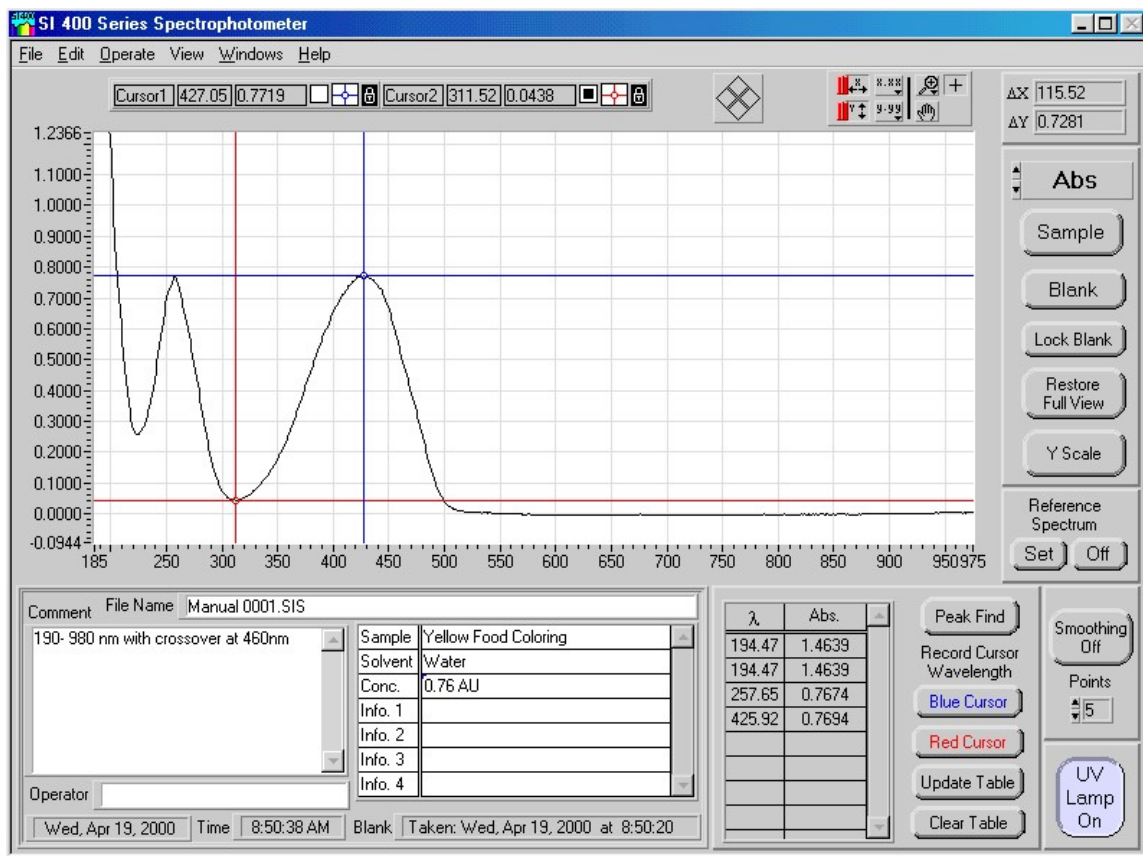


Figure 7-1 Main front panel

Another type of control on the front panel is used to select between a fixed set of options. This type of control will always have selection arrows on the left side of the control. For example, there is a control on the right side of the graphical output display that is used to select the display mode between absorbance, transmission, reflectance, intensity, and blank intensity. When the mouse is positioned over this type of control and the left mouse button pushed, the different possible choices will appear on the screen. The selected choice is highlighted. To change the selection, move the mouse forward and backward until the desired option is highlighted, then release the mouse button.

There are times when numerical information or text must be entered with the keyboard. An example is entering comments for a spectrum. For these types of controls, position the mouse pointer over the control and press

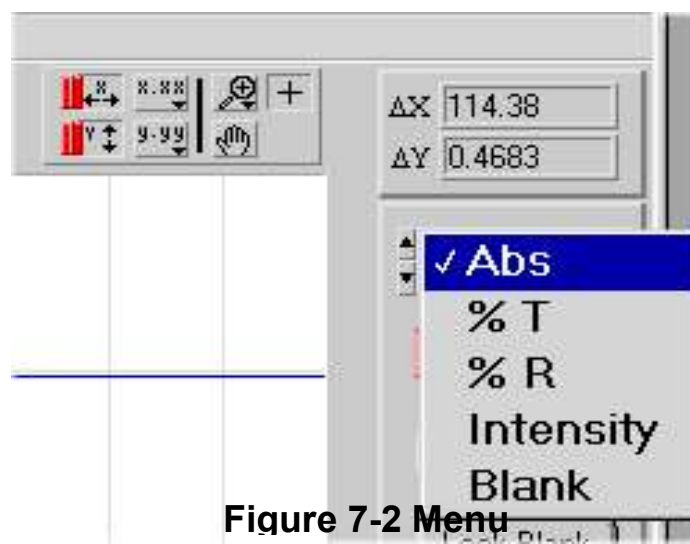


Figure 7-2 Menu

the left button. A cursor will appear, and you can enter information in the area provided much as you would with a word processor. The delete key will delete text, the left, right, up, and down arrows will move the cursor, and other keys will enter text. When you are finished entering information, do not hit the return key, but just move the mouse pointer to another location and operate another control.

7.2 Graphical Displays and Associated Controls

Graphical displays are used in several of the SI400 application front panels to plot two-dimensional data. For example, in the main front panel a graphical display is used to present absorbance, transmission, reflectance or intensity information as a function of wavelength. There are several types of controls used to facilitate panning, zooming, and positioning of cursors that are common to all of the graphical displays in the SI400 application.

All controls specific to the graphical displays are positioned immediately above the display (Figure 6-3 Graphical Display).



Figure 7-3 Graphical Display

Located just above the graph on the left side are indicators for the X and Y position (Figure 6-4 Cursor Display) for Cursor 1 (blue cursor) and Cursor 2 (red cursor).



Figure 6-4 Cursor Display

There is also a small square indicator that changes from white to black to indicate which cursor is currently enabled. An icon consisting of the cursor intersection within a small white box is a control that allows changing various cursor display attributes such as a cursor or no cursor. Clicking on this will bring up a menu of selections. An icon that appears as a padlock to the right of the attribute control is a control that forces the cursor to be constrained to the displayed data points, or free to move anywhere on the display.

Continuing to the right are two indicators labeled ΔX and ΔY that display the distance between the red and blue cursors.



Figure 6-5 Cursor Difference Display

The last control associated with the cursors is a four point diamond shaped icon (Figure 6-6 Cursor Single Step Movement). Pressing on the points of the diamond moves the selected cursor by single increments.

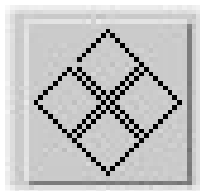


Figure 7-6 Cursor Single Step Movement

To the right of the cursor indicators and controls are a group of controls for panning, zooming, and auto-scaling (Figure 6-7). The two controls furthest left consist of buttons labeled X and Y, and an associated red colored slide switch. The buttons will cause the display to be immediately

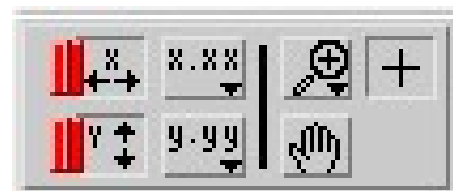


Figure 7-7 Graph Control panel

scaled to the data being displayed when pressed. The red colored slide switches, turn auto-scaling on or off for X and Y dimensions. If auto-scaling is turned on, the display scale will be adjusted to the data whenever the display is updated (such as when a new spectrum is acquired). When auto-scaling is turned off, the display scale will remain constant during data acquisitions. Two controls to the right of the scaling controls labeled X.XX and Y.YY allow the selection of the format and precision of the numbers displayed on the axis from a menu.

The next three controls, or tools, to the right of the scaling controls are used for pan, zoom, and cursor movement. The first of these tools has an icon that looks like a magnifying glass. Pressing this control brings up a menu of 5 different zoom modes, and a "undo zoom" selection (Figure 6-8 Graph Zoom Controls). Positioning the mouse pointer over the desired zoom mode and releasing the mouse button makes the zoom selections. Once the selection is made, positioning the mouse cursor in the area of the display to be zoomed, and pressing the mouse button performs zooming. The zoom mode selections include zoom to a selected X axis window, Y axis window, X and Y axis window, and zoom in or out around a central point. The pan tool looks like a hand, and when it is selected, the mouse cursor also appears as a hand in the graphical display region. Press the mouse button and the hand will "grab" the display. While holding the mouse button down, the display can be moved to pan to any desired region.

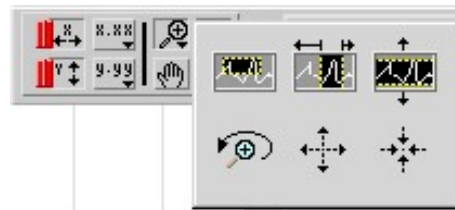


Figure 6-8 Graph Zoom Controls

Zooming can also be accomplished by directly editing the max/min labels on the axis of the graph. Clicking on the max or min label on either axis and editing it to the desired value does this. The tic marks will automatically be adjusted.

The cursor positioning tool that looks like a cross and when it is selected the mouse cursor also appear as a cross within the graphical display region. The cursors can be moved with the mouse by dragging them (press and hold down the mouse button while the mouse is over the cursor to select and move the red or blue cursor in the display). The coordinates of the cursor are displayed in the indicators in the top left of the graphical display as discussed previously.

The last control associated with the graphical display is the "hide grid/display grid" control that is accessed from the **View** pull down menu. Selecting the **Grid** item will either hide or display grid lines on both the graphical display and hard copy output depending on if the item is checked or not. If Auto-Scale is on when the grid state is changed, the display will re-scale.

7.3 On-Line Help

An on-line help feature is available to explain the purpose and functions of the various controls and indicators on all front panels except the previously described controls associated with the graphical displays. A help window is opened by selecting "show help" item from the **Help** item on the menu bar or with the key combination **Ctrl + H**. The help window will contain a description of the control or indicator that the mouse cursor is currently over. To view a description of a control or indicator, simply position the mouse cursor over the item of interest.

7.4 Function Keys

Using keyboard function keys in addition to the mouse point and click method can perform some operations. The function key assignments for these operations are noted both in this manual under the description of the operation and in the on-line help descriptions.

7.5 Setting Baud Rate and COM Port for the Host Computer

With a basic understanding of the software, the next step is for the computer to communicate with the spectrophotometer. When running for the first time after installation, the COM port and its baud rate on the host computer is set using the SI400 application. Start the application as described in Section 7.1, and select the **Configure** item in the Operate menu (Figure 6-9 Operate pull down menu) to make the **Configure** dialog box appear (Figure 6-10 Configure Dialog). In this box are controls labeled

Serial Port and Baud Rate, Serial Port Type, and buttons labeled Save and Close and printer type section (there are also two additional buttons for adjusting the lamp intensities that are disabled until the spectrometer has been initialized and are described later). To change the serial port selection, point to the Serial Port control and press and hold the left mouse button. A group of options labeled COM1 through COM4 will appear. While continuing to hold down the mouse button, slide the mouse up or down until the desired selection is highlighted. Release the mouse button to make the change take effect.

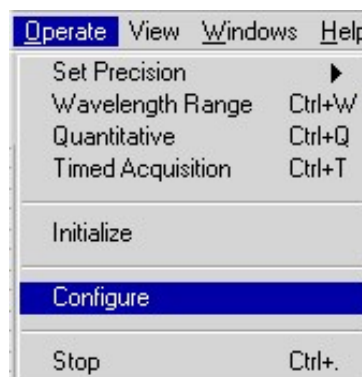


Figure 7-9 Operate pull down menu

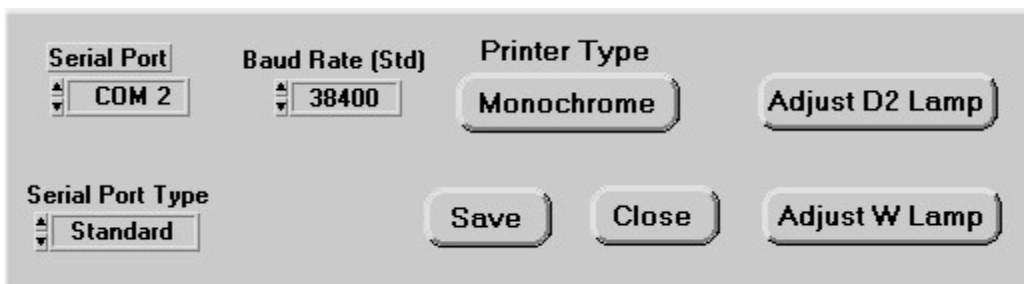


Figure 7-10 Configure Dialog

Note that the selection of a non-existent or unusable COM port is possible at this time. If this occurs, an error message will not be generated until the first attempt is made to communicate with the spectrophotometer during initialization.

A new baud rate is selected in the same manner as the COM port selection. After the COM port and baud rate selections have been made, press either the Save button to make the changes permanent, or the Cancel button to nullify all changes. When the Save button is pushed, the configuration changes are saved to a disk or hard drive. This file is read by the SI400 application when it is started, thus the configuration changes will be in effect any time the application is used in the future until it is intentionally changed again.

7.6 Initializing the Spectrophotometer

Once the communications parameters have been set up, we are ready to initialize the instrument. Spectrometer initialization involves communicating with the spectrometer to determine its status, then sending the initialization file. This must be performed every time power is cycled to the instrument. The communications link is used for the first time during initialization, thus if any communications problems are present they will become evident at this point. The initialization process is started by selecting the **Initialize** item located in the **Operate** menu (Figure 6-9 Operate pull down menu). If the communications link has been established, the file transfer will occur (taking ~2 seconds), and the spectrometer will be ready for use. Evidence of successful completion of initialization can be seen on the main screen. The button in the upper right corner labeled **Blank** will change from a nearly invisible light gray to a normal dark gray color. The tungsten lamp shutter in the spectrophotometer will also click as it opens and closes which may be used as an audible indication of successful initialization. If the wavelength range requires the UV lamp, a dialog box will appear asking if the user wants to start the UV lamp at this time. If the Initialization is not successful, see the Troubleshooting Section 16.1

8. Instrumental Procedures

This section will provide some basic instrumental procedures for acquiring absorption spectra in the main VI panel and the use of fiber optic accessories.

8.1 Fiber Optic Dip Probe

The standard fiber optic dip probe supplied with the 400 Series instruments consists of two (model 410/430 probe) or three (model 420/440 probe) fibers that converge at a point at the probe tip. Light from the lamps travels through the solution to a concave second-surface mirror, and is reflected back into a fiber that conducts the light to the spectrograph. The path length in solution is approximately 1 cm. The dip probe body is fabricated from type 316 chemically resistant stainless steel. The fiber optics and mirror are fused silica. A chemically resistant epoxy is used to attach the various components to the probe body. The protective sheath for the fiber optics is a steel monocoil clad with a PVC coating.

The probe should be immersed only for short periods of time (~10 sec.) in:

- 1) strong acids and bases
- 2) concentrated weak organic acids (such as concentrated acetic acid)
- 3) halogenated organic solvents

Rinse the probe well when it is removed from the above solvents.

8.2 Using the Standard Dip Probe

The probe should now be ready to use. The lamp intensities were adjusted at the factory but it may be necessary to adjust the tungsten and deuterium lamp intensities before the system is fully operational. If an error message appears when a blank is acquired indicating that the light source intensity is too high or too low, refer to Section 14.1.

The optical design of the standard dip probe dictates that it be used with solutions of very similar refractive index. The blank solvent should therefore match the solvent used for the standards and unknown solutions. It is not possible to use air as a blank.

When the probe is first immersed into a solution, a small bubble may form on the conical fiber tip. Briefly stir the solution with the probe to dislodge any bubbles. When the probe is extracted from a solution, a small drop of solution will remain on the mirror. This can be easily removed by tapping the side of the probe gently

on a bed of absorbent paper (such as Kimberly-Clark Teri or Kimtowels) placed on the counter top. The probe should be rinsed between samples in the blank solvent, and any wash solution also removed by tapping the probe.

8.3 Acquiring Absorption Spectra

After the instrument has warmed up (for best results, use one hour warm-up period), place the dip probe into the blank sample and press the **Blank** button. A brief shutter click-click will be heard as a short exposure is taken to determine the optimum exposure time. After this there will be a delay while a “dark” spectrum is taken without opening the shutter. This will be followed by a longer click---click of the shutter while the blank spectrum is taken. This dark spectrum is automatically subtracted from the blank and sample spectra. If both UV and visible regions are being used, this procedure will automatically be performed with the deuterium lamp as well.

After the blank has been taken, the **Sample** button will be enabled. Clean the probe as described above and place it in the sample to be measured. Press the **Sample** button to acquire the spectrum that will be displayed in the graph on the Main front panel. When a new spectrum is taken, the **Sample** button and the file name text box turns red indicating that the current spectrum is not saved. Once the spectrum has been saved the Sample and file name text will return to gray. Information about the spectrum can be entered into the File Information controls in the lower left portion of the VI panel. This information is saved with the spectrum. When the **Save** item is selected from the File Menu, a dialog allows the file name to be edited and a path to be selected for saving the data.

For more details about the operation of Main VI functions, see Section 9.

8.4 Cuvette Cell Holder

For measuring spectra of solutions using standard 1 cm pathlength cuvette cells or round ½” tubes, a cell holder accessory is available. The cuvette with the blank solution is placed into the cell holder and a blank is taken followed by a sample solution. If disposable cuvettes are being used, it is important to be aware of a mark that is normally at the top of the cuvette. This mark indicates the direction that the light should pass through the cuvette and for best results, the cuvette should be placed so that the mark is always facing in the same direction. Some quartz cuvettes also have direction indicators.

8.5 Fiber Optic Reflectance Probe

The reflectance probe is used to obtain the spectra of solid samples that are opaque. There is a collar on the tip of the probe that is used to set the distance

of the fiber optics from the sample. This is necessary so that the reflected light can enter the collection fiber. A blank should be taken using a white surface that is similar in texture to the sample. The user should be aware that some white papers have a UV dye in them that will absorb UV light and emit in the blue spectral region. This type of paper can cause problems when used as a blank. A piece of PTFE Teflon provides a good diffuse reflector for use as a blank when doing reflectance measurements.

8.6 External Shutter and Lamp Connectors

The SI430/440 spectrometer has connectors for both an external shutter and an external lamp. These connectors enable the use of optional accessories adding more flexibility to the instrument. If an external shutter was not included with your instrument and is needed, call S.I. Photonics and one can be provided. Note that the outputs on these connectors are designed to work with S.I. Photonics accessories. Please consult S.I. Photonics technical support before connecting any other devices to these connectors.

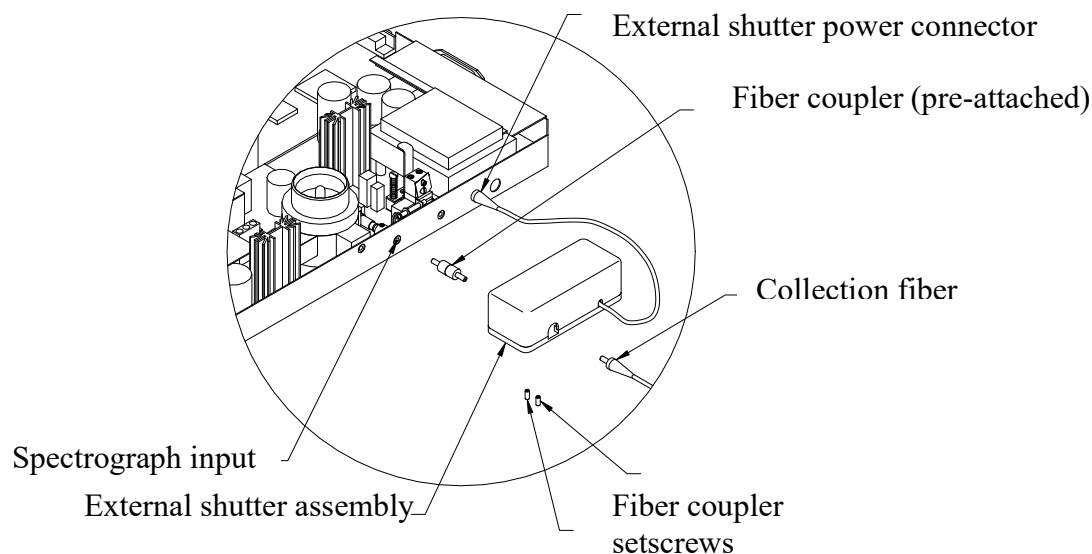


Figure 8-1 Setup of external shutter

8.6.1.1 External Shutter Connector

The external shutter connector is positioned to the right of the tungsten lamp output (see Figure 7-1 Setup of external shutter) and is the smaller of the two external connectors. The external shutter is used with an external lamp, radiometry measurements, and with integrating spheres.

8.6.2 External Lamp Connector

The external lamp connector is to the right of the shutter connector. When this connector is used, the internal lamp is disabled and a constant current is supplied at the connector. The external lamp connector is used with the integrating sphere accessory.

8.7 Integrating Sphere Accessory

The integrating sphere is used to provide diffuse illumination of samples that are difficult to illuminate properly with the reflectance probe.

8.7.1 Attaching Integrating Sphere

A lamp is mounted on one of the sphere ports at 90° to the sample port and there is a baffle to block direct illumination of the sample. The Power connector coming from the sphere for the lamp plugs in at "External Lamp". The collection fiber is mounted opposite the sample port. The port for the fiber will have a plug in the hole during shipping. Remove this plug by loosening the setscrew in the side of the port. Insert the fiber optic cable and tighten the setscrew. The collection fiber must be mounted to the external shutter, which is mounted to the spectrophotometer as described above. The sphere is mounted on a post above the sample area. A blank must be taken as in the reflectance measurement discussed above, followed by the sample measurement.

8.7.2 Integrating Sphere Operation

The sphere has 4 ports; lamp, fiber, reference and sample. The smaller ½" port is for the lamp. The port opposite the lamp can be used as a reference port to improve sampling accuracy. The sample port is 90° from the lamp port and has a baffle to prevent the light from the lamp directly illuminating the sample. The fiber port is 180° from the sample port.

Reference port: A small amount of the light reflecting off of the object placed in front of the sample port will reflect back into the sphere, bounce around and then eventually strike the sample again. Since this light has already been filtered by the sample it is no longer white light and can cause a minor effect on the measurement if the sample is very dark. To eliminate this effect the blank measurement is taken with the sample or similarly colored object in the reference port. The white reflectance standard in the sample area is then switched to the reference port during sample measurements. The user should determine if this minor effect has a relevant impact on their measurement accuracy.

Lamp port: The sphere has an internal tungsten lamp similar to the one in the instrument. The lamp has a lower color temperature and longer life than the standard lamp used in the instrument. The Spectral part number for this lamp is 1662. The lamp will be very hot, wait for it to cool before replacing. Unplug power to the lamp before replacing. To change the lamp, loosen the two tiny setscrews holding the lamp housing onto the sphere. Unplug the lamp and replace it. Do not touch the lamp with your fingers. The oils from your fingers will damage the lamp. If the lamp is touched clean with alcohol before using.

Fiber port: The fiber port is where the light is collected and transferred to the spectrometer. For large objects longer fiber optic cables can be used to increase the distance of the integrating from the instrument.

Sample port: The fiber numerical aperture and the spectrometer optics limit the sample area to about a 3/4 inch diameter at a 4 inch distance. Smaller spot sizes can be achieved with the use of the port reducers. The sample should be placed as close to the sample port opening as possible. The sample should also be placed at the same distance each time. The sphere is shipped with a general-purpose stand but most samples are best measured with custom holders to position specific samples or to place the sphere on a flat object.

9. The SI400 Main Front Panel

The main front panel controls are used for configuring and initializing the instrument, general data acquisition, examining spectra, and saving spectra. It is also used to gain access to other specialized panels such as the quantitative analysis, timed spectral acquisition, peak find and multiple spectra display front panels.

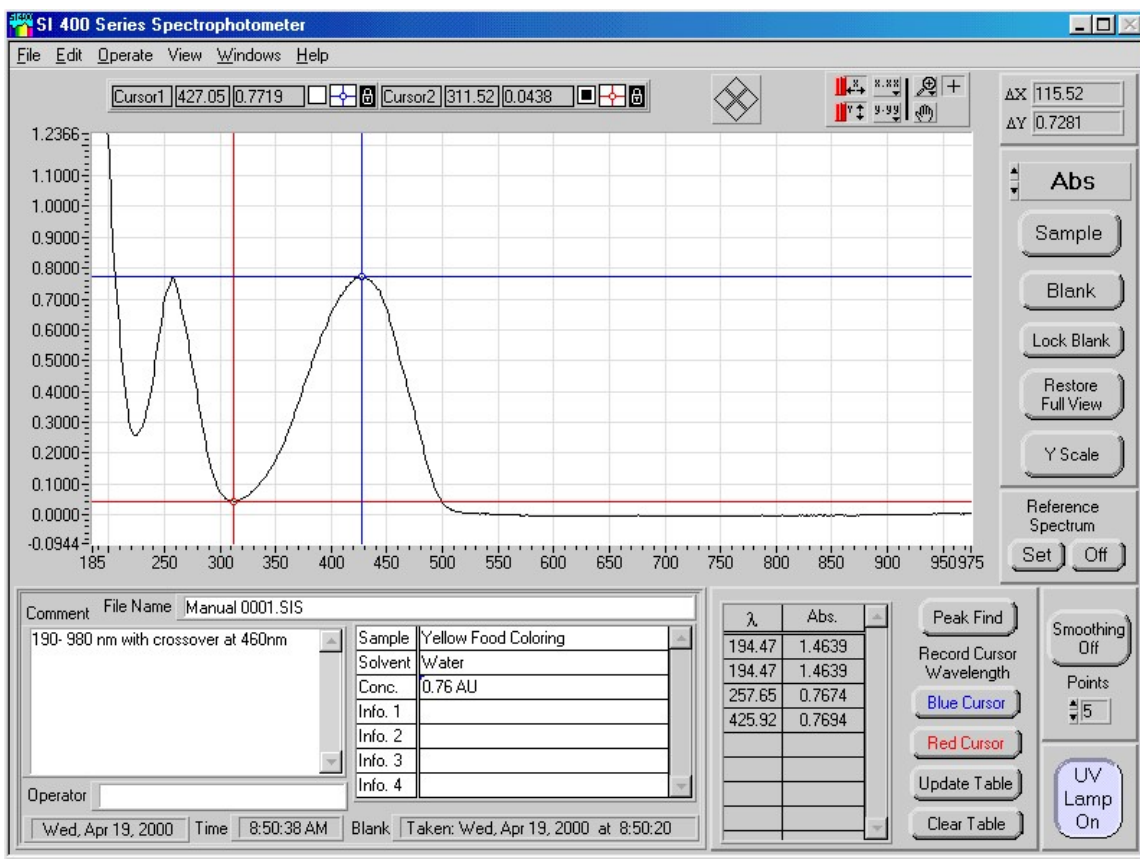


Figure 9-1 Main front panel

9.1 Graphical Display

The graphical display on the main front panel is used to present absorbance, transmission, reflectance, sample or blank intensity as a function of wavelength. The controls use to manipulate the display are discussed in Section 7. The type of data presented on the graphical display can be changed using a control located near the upper right corner of the graph. Options are:

Abs - Calculates and displays absorbance of sample referenced to blank.

% T - Calculates and displays % transmission of sample referenced to blank.

% R - Calculates and displays % reflectance of sample referenced to blank.

Intensity - Displays raw light intensity of sample in arbitrary units.

Blank - Displays raw light intensity of the blank in arbitrary units.

The current display mode is always indicated on the control.

a. Main Front Panel Controls

The following is an alphabetical listing of the Main front panel controls.

Blank - Acquires and stores spectrum of blank sample for calculation of absorbance and % transmission relative to Sample spectrum. Data is stored until the wavelength range is changed or a new blank is acquired.

Function key F2

Blue Cursor - Adds an entry into the wavelength column of the data table at the current position of the blue cursor.

Function Key F9 (records blue cursor position and updates table)

Clear Table - Clears the data table.

Configure – Option of the Operate pull down menu, used to set the COM port and baud rate for communication with the spectrophotometer and to access the lamp adjustment screens.

Export Files – From the **File** menu exports a single file or a group of files that have been saved on the disk to an Excel or delimited ASCII text spreadsheet file. Select the **Export** function from the **Files** menu and a window will open (Figure 9-2 Directory Selection) asking the user to select the directory or folder containing data files to be exported

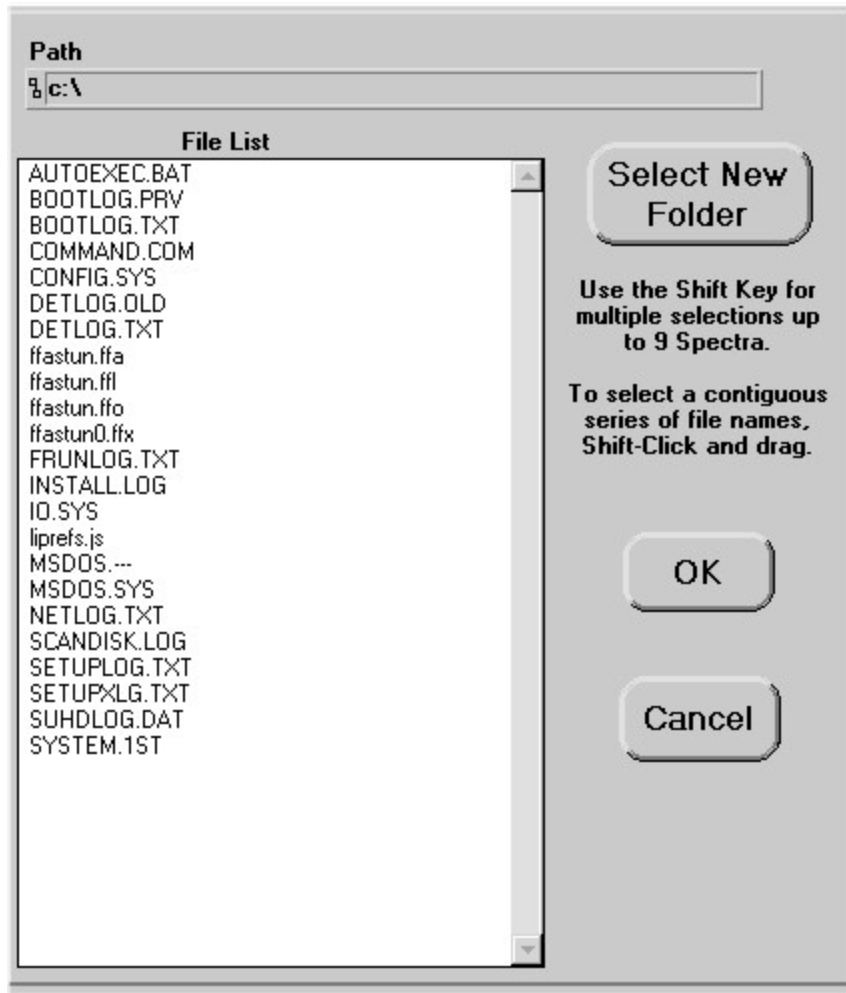


Figure 9-2 Directory Selection

In the new window click on Select New Folder, to open another window directory and open the folder where the saved files are located. Highlight the folder and click on Select Cur Dir. (Do Not click on Open) See Figure 9-3 Selecting File Folder.

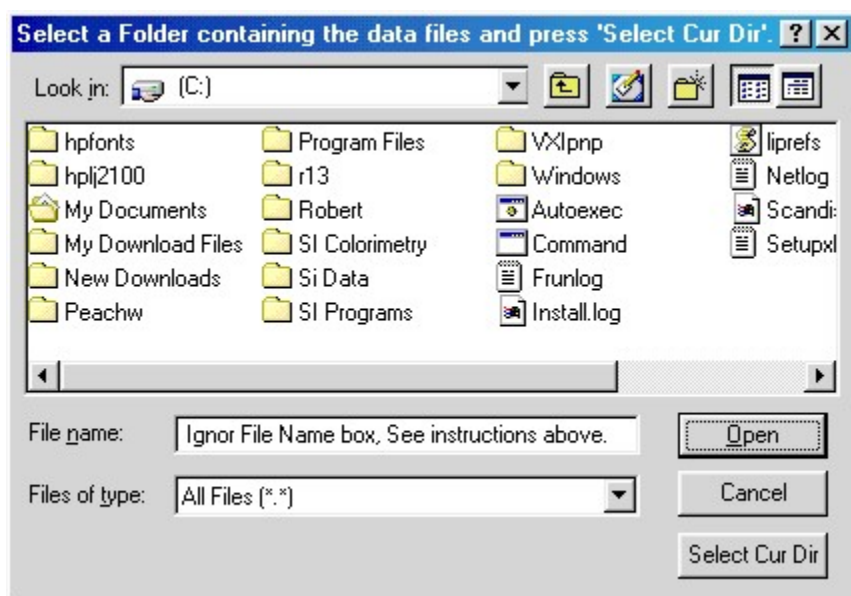


Figure 9-3 Selecting File Folder

The next screen (Figure 9-4 – Selecting Individual Files) lists all the spectrum in the selected folder. The user can select one spectrum to be exported or a group up to 9 spectra. To choose more than one file, use the shift key for multiple selections. For spectrum in a contiguous series of file names Shift-Click and drag the mouse. Click OK.

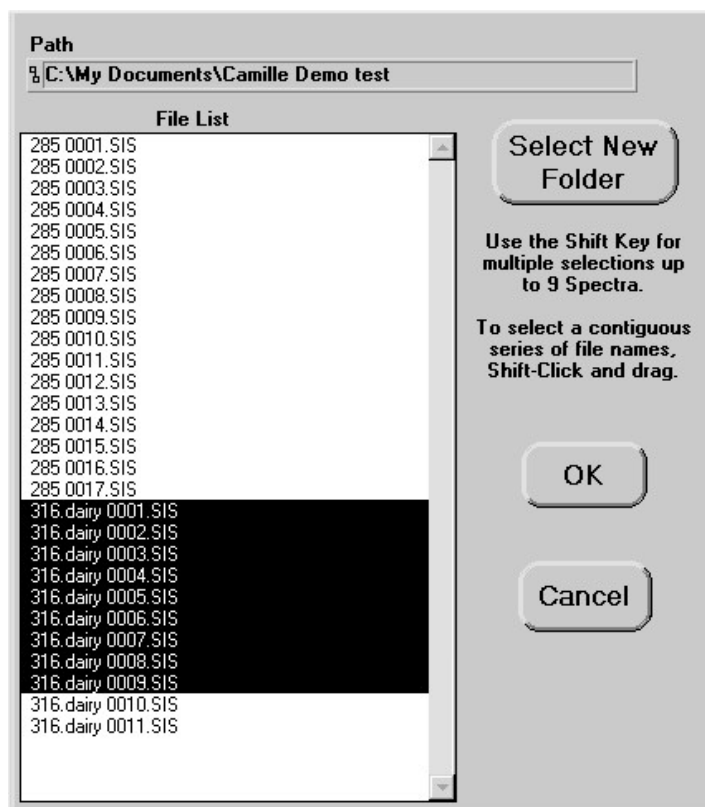
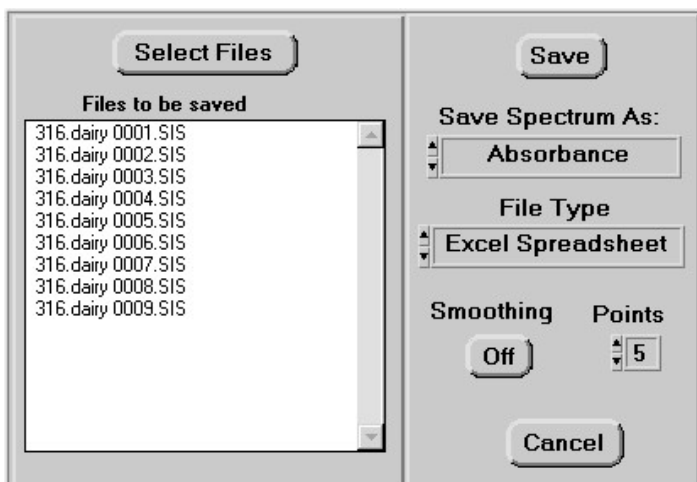


Figure 9-4 Selecting Individual Files.



Another window opens and list the selected files and allows the user to choose the type of data to export, the type of file format, and whether to smooth the data.

Figure 9-5 Selecting File Format

The last panel in the export function pops up. This window saves the files in the new format in the location the user has selected. The exported data must have the same wavelength range since the first column contains the wavelength values and each successive column contains data for each selected file. Also at this point the user can complete the export process by pressing the **Save** button, or **Cancel** the operation. When the **Save** button is pressed, a dialog opens up (figure 9-6 Select file name and path for exported data) that allows the user to choose the path and file name for the exported data.

Function Key Ctrl+E

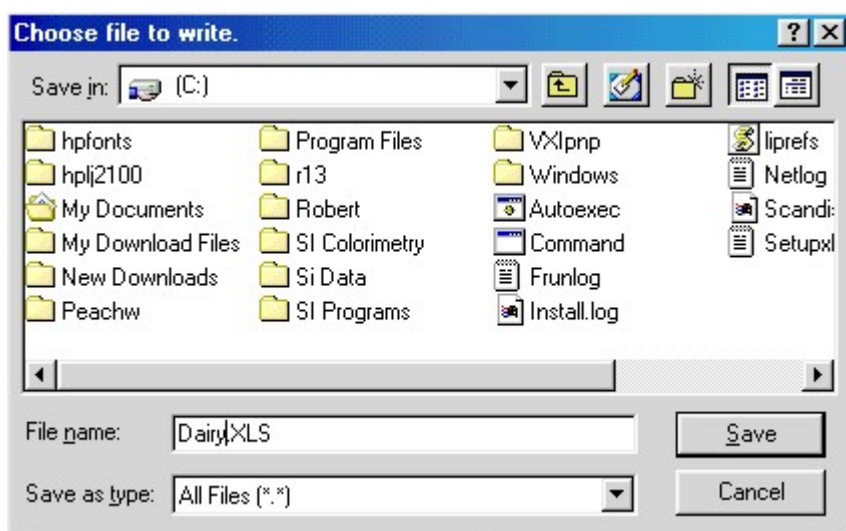


Figure 9-6 Choosing Files to Save.

File Name Filter - limits the sort criteria when retrieving files from the disk.

Grid – From the View menu allows user to add or remove the grid lines in the spectr Figure 9-6 Select file name and path for exported data a display.

Function Key Ctrl+G

Initialize – Found in the Operate menu, Initializes the spectrophotometer. This function needs to be performed only on start up or if power to the spectrophotometer has been turned off.

Lock Blank - This button will disable the **Blank** button until the lock is released by is pushing this button again. This prevents accidental acquisition of a new blank.

Open - Opens a data file created with the **Save** item in the File menu. The spectrum in the data file, along with all other file information, is displayed.

Function Key F6 or Ctrl+O

Multiple Spectra – from the **View** menu opens a new front panel to view up to 10 spectrum at once. Refer to section 13, the Multi Spectrum Display instructions for command summary on this panel. Push the **Close** button to return to the Main front panel.

Auto Add to Multiple Spectra – automatically places the newly acquired spectra into Multi Spectrum Display panel. The default is On.

Function Key Ctrl+M

Peak Find – Opens a new front panel for peak and valley analysis. Refer to the Peak Find instructions in section 12 for command summary on this panel. Push the **Close** button to return to the main front panel.

Function Key F4

Precision – Access through the Operate menu

Low - This mode is useful for fast measurements in the 0.05 to 2.00 absorbance range. The array detector is read 2 times and averaged.

Normal - This mode is adequate for most measurements in the 0.05 to 2.00 absorbance range. The array detector is read 20 times and averaged.

High - Increases precision of measurements by averaging 100 reads of the array detector. Use only when the highest precision is necessary. This mode increases the time to acquire both sample and blank by approximately a factor of 5 compared to Normal mode.

Print Graph & Info - Outputs spectrum, comments section and data table displayed on the panel to the selected Windows output device.

Function Key Ctrl+P

Print Only Graph - Outputs spectrum only

Quantitative –Accessed through the Operate menu, opens a new front panel for quantitative analysis. Refer to the quantitative analysis instructions for command summary on this panel in section 10. Push the **Close** button to return to the main front panel.

Function Key Ctrl+Q

Red Cursor - Adds an entry into the wavelength column of the data table at the current position of the red cursor.

Function Key F10 (records red cursor position and updates table)

Reference Spectrum – Allows for two spectra to be viewed and one is designated as a reference. There are two controls to turn on or off displaying the reference spectrum, and one to turn a displayed spectrum into the reference spectrum.

Set - Changes currently displayed (black colored) spectrum into the reference (red colored) spectrum.

Function Key F11

On/Off - turns on and off (depending on current state) the display of the reference spectrum and activates the Set button. This button also controls whether or not the reference spectrum is printed on hard copy.

Function Key F12

Restore Full View - – Displays original spectrum to full scale

Function Key F3 or Ctrl + F

Sample - Acquires a spectrum of a sample. Performs a calculation for the display mode selected. A blank must be acquired before the **Sample** button becomes active.

Function Key F1

Save - Saves the displayed spectra, comments and data table shown on screen to disk. Data is retrieved with the **Open** item. The file formats default is compatible with the SI400 application only.

Function Key F7 or Ctrl+S

Save As – Saves the spectrum in different format then the *.SIS default. *These files can not be open in the SI 400 application once saved in the file formats below.*

Text - Save spectrum data into comma delimited text file.

Excel – Saves spectrum into Microsoft Excel format.

Auto Save – Automatically saves the spectrum to a chosen file after sample is taken. The default is off. To switch on, click **Auto Save On**. A new window will open to select path (Figure 9-7).

Auto Save As – Automatically saves sample spectrum as the default *.SIS format or can be selected to save as *.text file. Select by checking the chosen option.

Save Setup - Saves the current settings of the program to be recalled when the spectrometer is initialized. The settings saved include the wavelength range, lamp crossover wavelength, the table of wavelengths to be monitored, current disk paths for data, precision setting, smoothing on/off and points, and the grid on/off.



Figure 9-7 Auto Save Path settings

Smoothing - Smooths the data that is displayed and that used for quantitative data analysis by a Savitzky-Golay quadratic-cubic algorithm with an adjustable number of points. This effectively increases the signal to noise ratio of spectral data at the expense of reduced spectral resolution. Data saved to disk files is not saved in smoothed form, but any file read from disk can be smoothed. The smoothing algorithm is described in J. Anal. Chem., 1964, 36, p. 1627.

On/Off - turns smoothing on or off. Only the currently displayed spectrum is affected. Once smoothing has been turned on any subsequently acquired spectra, or data files read from disk will be automatically smoothed.

Points - varies the number of points in the smoothing algorithm. As the number of points is changed, the current spectrum displayed is dynamically updated with the new smoothing parameters.

Stop - Used to clear errors, also halt operation of the program. This item is located in the Operate menu. Restart by clicking on the arrow in title bar.

Function Key Ctrl+.

Timed Acquisition – Accessed through the Operate menu opens a new front panel for repetitive timed spectral acquisition. Refer to section 11 the timed acquisition section for command summary on this panel. Push the **Close** button to return to the main front panel.

Function Key Ctrl+T

Update Table - Updates the absorbance, % transmission, % reflectance or intensity fields in the data table. Used when wavelengths are manually entered in the data table rather than using the blue cursor/red cursor buttons.

UV Lamp (model 420 & 440 only)

On/Off - Turns on or off the deuterium lamp. The lamp power supply requires approximately 20 seconds to start the lamp discharge. The lamp should be allowed to warm up for at least 20 minutes and preferably 1 hour to reach maximum stability. Baseline drift will be observed if the lamp has not had time to stabilize.

Wavelength Range - From the Operate menu opens a dialog box which allows the operator to change the wavelength range of the spectrum that will be acquired and the lamp crossover wavelength. Limiting the range to wavelengths of interest will speed up data transfer and reduces the size of saved files. The minimum wavelength is 190 nm for Model

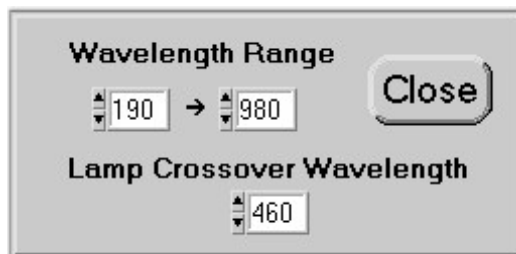


Figure 9-5 Wavelength range Window

440 and 350 nm for Model 430. The maximum wavelength is 980 nm for both models. The lamp crossover wavelength (Model 420 & 440 only) is the wavelength where the change from the deuterium to tungsten lamp is made. The default in the initial setup is 460 nm. This gives the best overall performance. The lamp crossover can be set from 350-800 nm. When it is set to >460 nm, sharp and intense structure in the deuterium spectrum near 485, 580, and 656 nm can cause errors in the acquired spectrum near those wavelengths. Also, due to the effect of the intensity scaling caused by these features, the overall precision can be adversely affected.

Key Function Ctrl + W

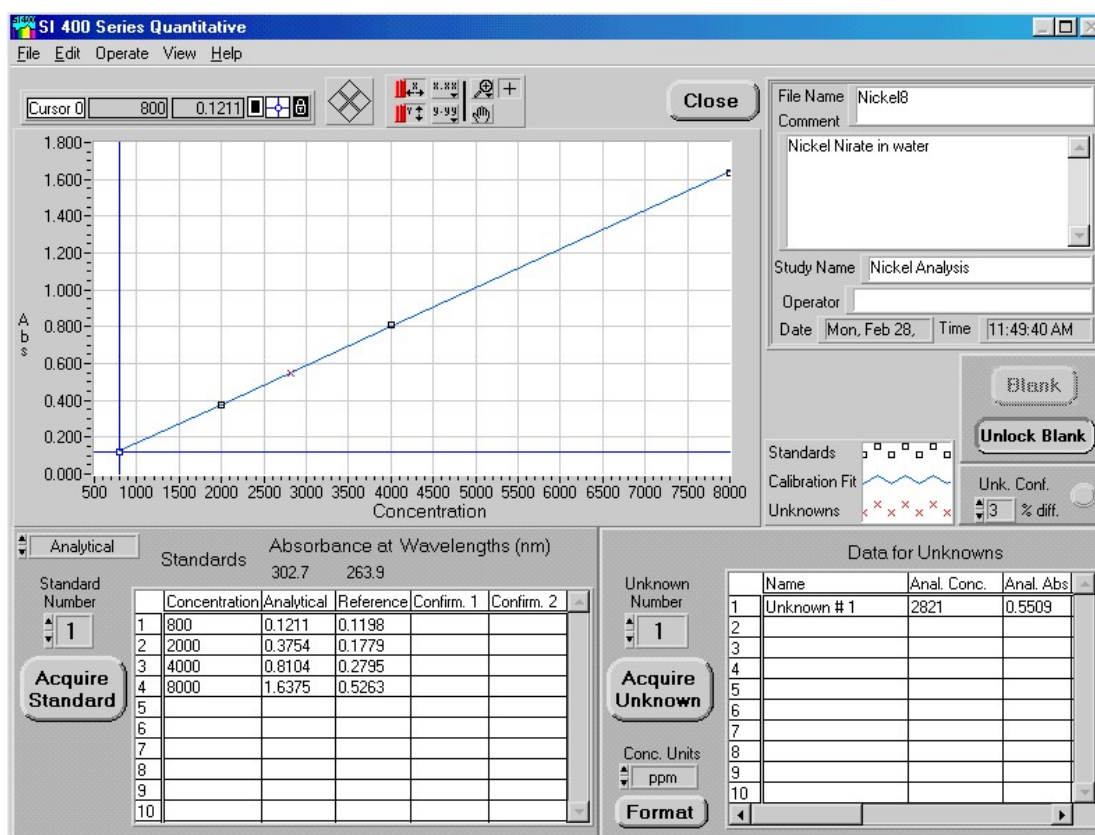
NOTE: A new blank must be acquired every time the range is changed. Once the desired wavelength range is selected, save the parameters by going to the File menu and clicking on **Save Setup**

Y Scale – adjusts the y-axis to fit the spectrum to fill the full display.

Function Key F5

10. The SI400 Quantitative Front Panel

The Quantitative front panel is used to acquire and analyze data for quantitative analysis of samples. Before opening the Quantitative front panel, it is best to take a blank spectrum and a sample spectrum (see Section 8.3) of a standard solution to facilitate the choosing of analysis wavelengths. It is opened by selecting the **Quantitative** option under the Operate menu in the Main front panel. Controls are available to acquire data from standards and unknowns, fit standards data to a curve, and determine concentrations of unknowns based on the standards data. The Quantitative front panel has a graphical display for analytical data and a working curve, a section for controls and data related to analytical standards and a section for controls and data related to unknowns.



10.1 Quantitative Analysis Front Panel

The standard number and concentrations for each standard are entered in the Standards Table. The absorbance for the standards at the analytical wavelengths and the confirmation wavelengths will automatically be entered into the rest of the table when the **Acquire Standard** button is pressed.

Description for the unknowns is entered in the Unknowns table. Concentration and absorbance results will be calculated and inserted when the **Acquire Unknown** button is pressed.

10.2 Graphical Display

The graphical display on the Quantitative front panel is used to present absorbance as a function of concentration information. The controls use to manipulate the display are discussed in Section 7.2. Data from the analytical wavelength, or confirmation wavelengths can be selected using a control located near the lower left corner of the display.

10.3 Quantitative Analysis Procedures

Once the Quantitative window is open, the procedure for doing an analysis is as follows:

1. Press **Selected Wavelengths** button to open the Quantitative Wavelengths Selection from the Operate menu (Figure 10-2) that will allow the selection of the wavelengths to be used for the analysis.

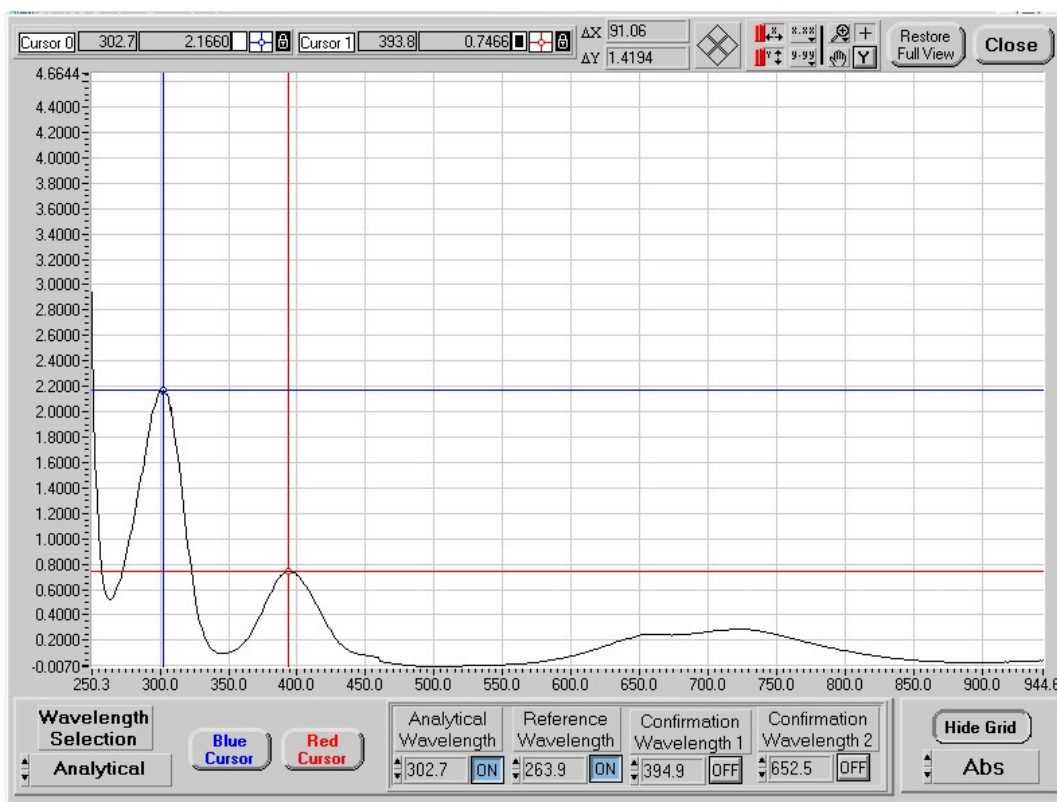


Figure 10-2 Quantitative Wavelengths Selection panel

The analytical wavelength is usually the wavelength of maximum absorbance for the sample. The reference wavelength is usually a point of minimum absorbance for the sample. When a reference wavelength is selected, the absorbance at this wavelength is subtracted from the absorbance at the analytical wavelength during the computations in the Quantitative front panel. The confirmation wavelengths are typically used for tests of purity or chemical interference in a sample. The wavelength is usually chosen in a region of appreciable absorbance, but removed from the analytical wavelength.

The selection of analytical, reference, and confirmation wavelengths may be made by typing in the value into the appropriate box. An alternate method is to position a cursor at the desired location on the spectrum in the graphical display. Select Analytical, Reference, Confirmation 1, or Confirmation 2 on the **Wavelength Selection** control and press the **Blue Cursor** or **Red Cursor** button depending on which cursor is being used. The wavelength of the cursor is entered into the selected item. *A spectrum of an analytical standard must be acquired in the Main front panel to use the graphical input approach.*

Each type of wavelength must be turned **ON** in order to be incorporated into the quantitative calculations.

2. There is an option to take a new Blank spectrum if it is deemed necessary. Press the **Unlock Blank** button to make the **Blank** button active and proceed with taking a new blank as described in Section 8.3. After the blank is taken, the **Blank** button is automatically locked to lessen the chance of it inadvertently being pressed.
3. Enter the concentrations of the standards into the Standards table. If there are more than ten standards, a scroll bar will appear on the right side of the table to allow the table to be scrolled down.
4. Next, acquire the spectra for the standards. When the **Acquire** button for standards is pressed, the data will be put into the table for the standard number indicated in the **Standard Number** control. This control defaults to 1 and will automatically increment to the next standard number. This control can also be used to manually set the next standard number to be acquired.
5. When all the standards have been acquired, select the **Calibrate** item in the Operate menu and the calibration curve (linear least squares fit) for the data will be calculated and displayed on the graphical display. At this point, the fit results can be viewed by pressing the **View Fit Results** option in the Operate menu.
6. Now we are ready to measure the unknown samples. Prepare the first unknown sample to be measured. A description can be typed into the unknown table in the Name column. The **Unknown Number** control selects

the unknown number in the **Unknowns** table for which data will be collected. This control is initially set to 1 and will automatically set to increment to the next number after a new spectrum is acquired. Data is acquired by pressing the unknown **Acquire** button and the results will appear in the **Unknowns** table. There are scrollbars on this table for both vertical and horizontal movement. The horizontal scrollbar allows viewing of the results for the Confirmation wavelengths.

Further details of the on the Quantitative front panel are given below.

10.4 Quantitative Front Panel Controls

Acquire - Two Acquire buttons are present, one in the standards section and the other in the unknowns section. These buttons acquire a spectrum for the data tables they are adjacent to. The standard and unknown numbers are chosen before the acquisition, then data is stored in the corresponding line in the table. For the standards **Acquire** button, the known concentration must be entered before taking the spectrum.

NOTE: Analytical, reference and confirmation wavelengths must be set before the button is active. See **Wavelength Selection**.

Function Key Acquire Standards F1

Function Key Acquire Unknowns F6

Blank - Acquires and stores spectrum of blank sample for calculation of absorbance and % transmission relative to Sample spectrum. Data is stored until the wavelength range is changed or a new blank is acquired.

Calibrate – Found in the Operate menu calculates the best fit function to standards data using linear regression.

Clear Standards - Clears all information entered into the standards table and accessed from the Operate menu.

Clear Unknowns - Clears all information entered into the unknowns table and accessed from the Operate menu.

Close - Closes the Quantitative front panel and opens the Main front panel. Data in the Quantitative front panel will not be lost.

Concentration units

Molarity - data units displayed as molarity (mol/L).

PPM - data units displayed as parts per million.

File Name Filter - Limits sort criteria when retrieving files.

Fit Results - Displays statistical data from linear regression for Analytical and Confirmation wavelengths and is found in the Operate menu.

Format - Sets the display preferences for concentration values . The units may be decimal, scientific or engineering. Number of decimal places displayed is also set here.

Function Key F11

Open - Retrieves a data file saved with the **Save** command from disk found in the File menu.

Print Graph - Outputs data in graph, comments section, standards table, and unknowns table displayed on the screen to selected output device. This selection is accessed in the File menu.

Function Key Ctrl+P

Save - Saves the displayed data and comments shown on screen to disk and accessed in the file menu. Data is retrieved with the **Open** item. The file format is compatible with the SI400 application.

Function Key Ctrl+S

Save as Text - Saves data as an ASCII tab delineated file for export to spreadsheets and data manipulation programs. Save as Text is accessed from the File Menu.

Save as Excel – Saves data as an Excel spreadsheet file for export later to perform data manipulations and accessed from the File menu.

Select Wavelengths – In the Operate menu opens a new front panel that displays a graph of a spectrum for the selection of analytical, reference and confirmation wavelengths for the quantitative analysis front panel. Refer to the Wavelengths front panel section 10.5 for a summary of the controls on this screen

Spectrum – Found under the View menu displays a spectrum at full screen size of the most recent spectra acquired.

Standard number - Changes the line number where data is inserted in the standards table when **Acquire** button is pressed. This number will automatically increment by one after each sample.

Unk. Conf. - Used to set the acceptable percent difference between analytical and confirmation concentration. A warning will be displayed if unknown concentration is out of tolerance.

Unlock Blank - This button will enable/disable the **Blank** button. This prevents accidental acquisition of a new blank.

Unknown number - Changes the line number where data is inserted in the Unknowns table when **Acquire** button is pressed. This number will automatically increment by one after each sample.

10.5 Quantitative Wavelengths Front Panel Controls

The following is an alphabetical listing of the Quantitative Wavelengths Front Panel controls (Figure 10.2 Quantitative Wavelength Selection panel).

Analytical Wavelength - Is a control where the analytical wavelength can be directly entered from the keyboard. There is also an on/off control to enable or disable the use of the analytical wavelength in the quantitative front panel.

Blue Cursor - Enters the wavelength into the selected control box (see Wavelength selection) at current position of the blue cursor.

Close - Closes the Analytical Wavelengths window and brings back the Quantitative screen.

Confirmation Wavelength 1 and **Confirmation Wavelength 2** - Are controls where the confirmation wavelengths can be directly entered from the keyboard. There is also an on/off control to enable or disable the use of confirmation wavelengths in the quantitative front panel.

Red Cursor - Enters the wavelength into the selected control box (see Wavelength selection) at current position of the red cursor.

Reference Wavelength - Is a control where the reference wavelength can be directly entered from the keyboard. There is also an on/off control to enable or disable the use of a reference wavelength in the quantitative front panel.

Wavelength Selection - Determines which wavelength type will be recorded when the Red Cursor or Blue Cursor button is pushed. Selections are:

Analytical
Reference
Confirmation 1
Confirmation 2

11. The SI400 Timed Acquisition Front Panel

The timed acquisition front panel is accessed by going through the Operate menu of the Main front panel and is used to acquire spectral information at fixed time intervals. The wavelength range over which spectra will be acquired must have been previously set using the wavelength range item on the Main front panel's Operate menu.. The panel that opens has a graphical display similar to the one on the Main front panel. An acquisition mode selector allows the selection of full spectrum acquisition, or discrete wavelengths. There are some changes in the front panel depending on the mode selected.

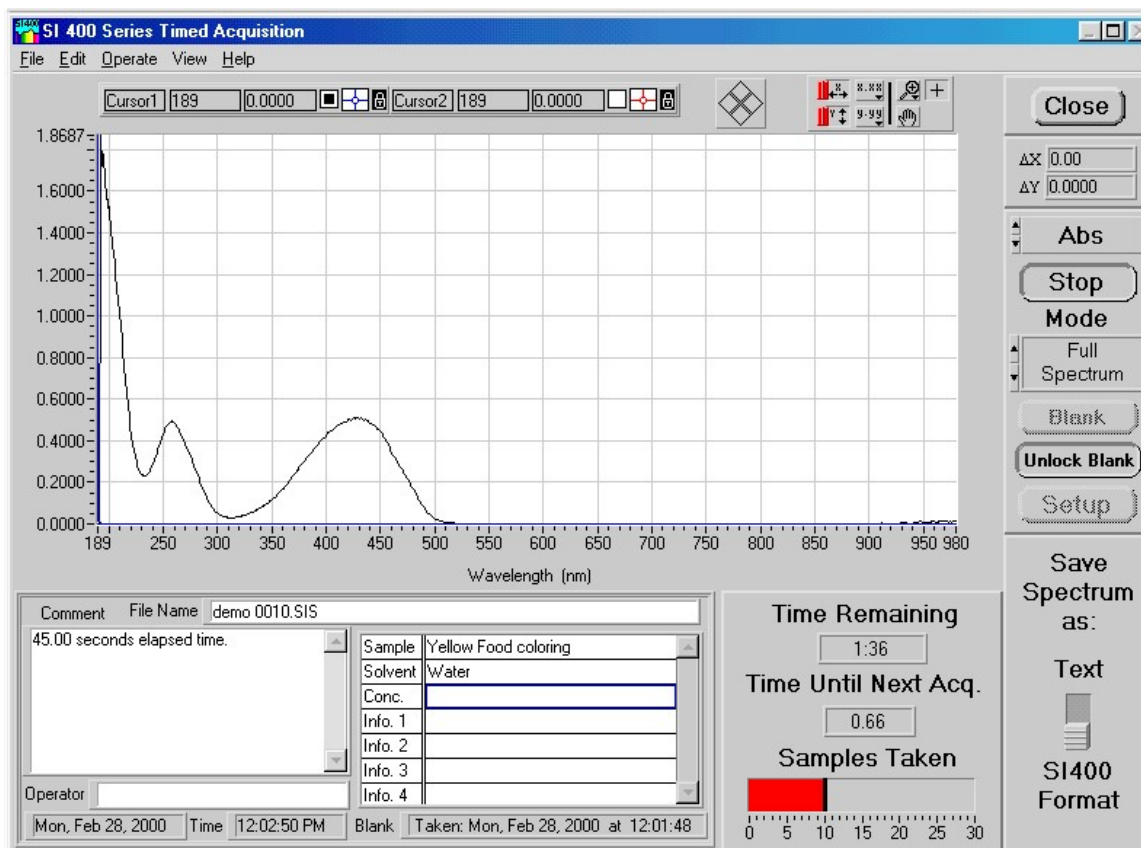


Figure 11-1 Timed Acquisition, Full Spectrum Mode

In full spectrum mode (see Figure 11-1), the spectra are automatically saved either as text files or in SI400 format as the user selects. The naming and path for saving the spectra is specified in the acquisition setup covered below. Opening and printing a spectrum taken with timed acquisition is performed in the Main front panel.

In discrete wavelength mode (see Figure 11-2), a table appears displaying the wavelengths to be monitored, along with a color assignment for that wavelength as it is plotted on the graphical display. Data acquired at discrete wavelengths can be viewed graphically as changes in absorbance or transmission as a

function of time. As many as ten discrete wavelengths can be simultaneously monitored.

An assortment of controls for saving, opening, and printing data files similar to that found in the discussion of the Main front panel appear on this Timed Acquisition front panel. Files from data acquired in the discrete wavelengths mode are incompatible with those acquired using the full spectrum mode, and can only be viewed from the Timed Acquisition front panel. There is an additional command on the discrete wavelengths mode panel for kinetics that is described below.

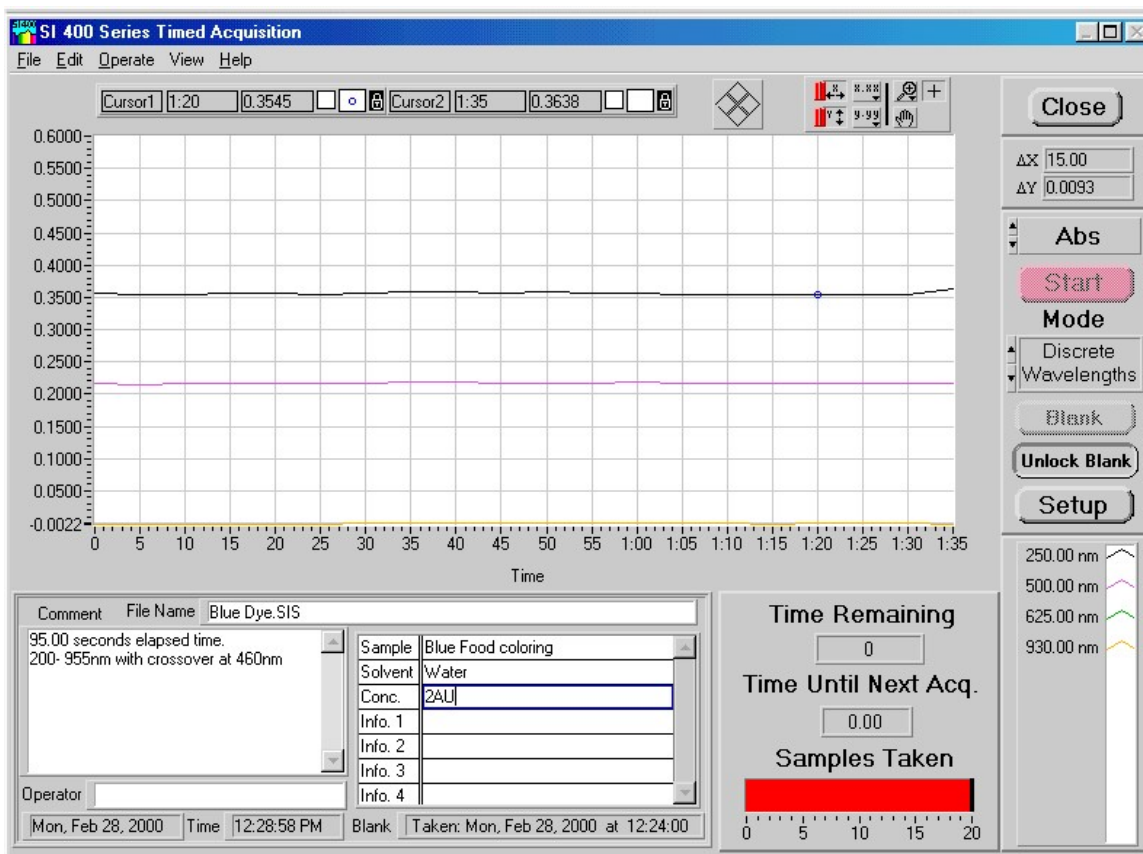


Figure 11-2 Time Acquisition, discrete Wavelengths Mode

11.1 Timed Acquisition Front Panel Controls

The following is an alphabetical listing of the Timed Acquisition front panel controls.

Close - Closes the timed acquisition front panel and re-opens the Main front panel.

File Name Filter - Limits sort criteria when retrieving files and is accessed from the File menu..

Kinetics – From the Operate menu opens a Kinetics front panel (Figure 11-3 Kinetics front panel) that allows the viewing of discrete wavelength data as a kinetic plot of raw data or a first order plot with rate constants for each wavelength measured. There is also an option to limit the plot display and rate calculations to the data that is between the two cursors.

Mode – Allows the selection of full spectrum or discrete wavelength acquisition modes.

Open - Accessed from the File menu retrieves a data file from the disk that was previously saved.

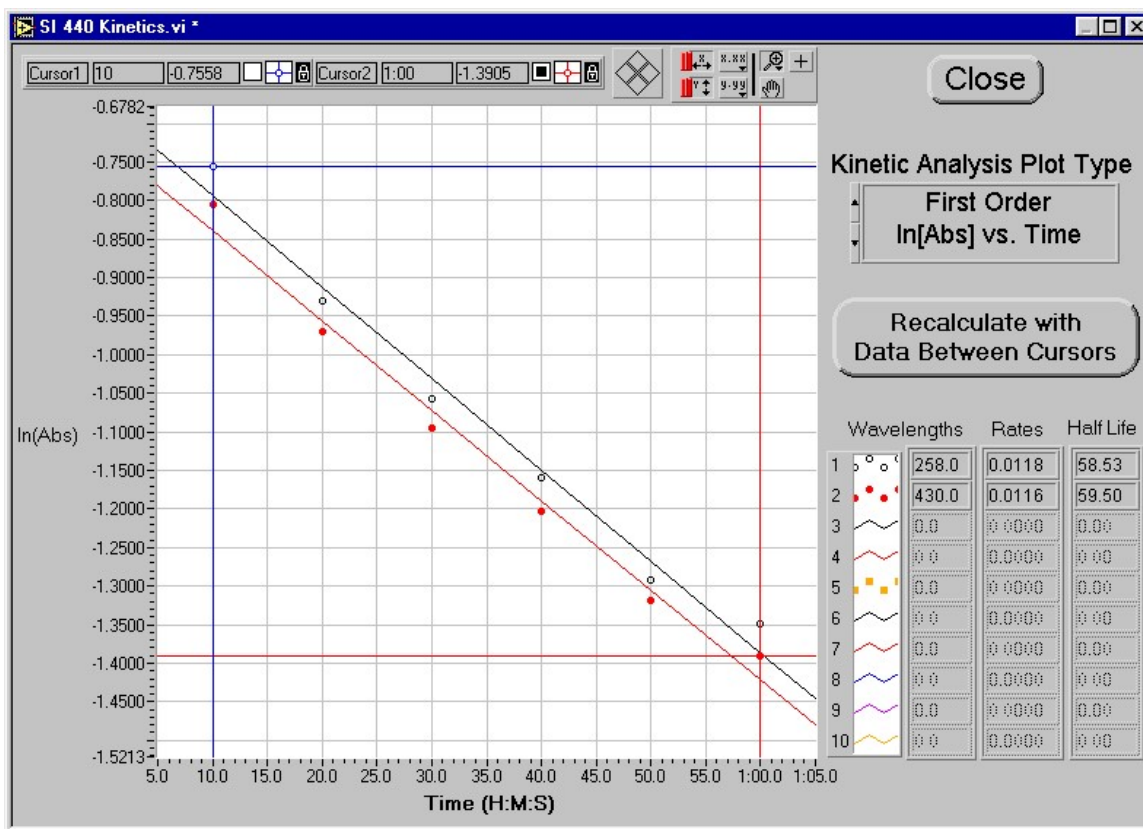


Figure 11-3 Kinetics front panel

Print - Outputs data in graph, comments section, and wavelengths displayed on the screen to selected output device. The **Print** command is found in the File menu.

Samples Taken - This indicator shows the number of data acquisitions completed. This number is updated as acquisitions are made.

Save – In the File menu, the Save item saves the displayed data and comments shown on screen to disk. Data is retrieved with the **Open** command. The file format is compatible with the SI400 application only.

Save as Text – From the File menu saves data as an ASCII tab delineated file for export to spreadsheets and data manipulation programs.

Save as Excel – From the File menu saves data as an Excel spreadsheet file for export later to perform data manipulations

Setup - Opens a dialog box (see Figure 11-4) where a number of setup parameters are selected. The dialog box changes depending upon which mode (full spectrum or discrete wavelength) is chosen. In full spectrum mode, the dialog box allows selection of the time interval between acquisitions, number of acquisitions, and the file information, and specific wavelengths of interest. The full wavelength spectrum is saved and if any wavelengths are entered into the Wavelength table, these wavelengths are also saved with the spectrum. When the spectrum is opened in the main front panel, the values at those wavelengths will be displayed in the Wavelength table on the Main front panel.

Full Spectrum:
Set the Time Interval, Number of Spectra (Acquisitions), and File Information.
Any wavelengths set will be saved with the spectrum and values will be displayed in the main front panel.

Ok

Time Interval Sec/Min: 5.00 Sec. ▼

Number of Acquisitions: 10

File Name is "Data Series ###.SIS"

Data Series: TEST

Start at #: 1

Select Folder: C:\My Documents

Clear

Wavelengths

375.00
400.00
500.00
600.00
625.00
630.00
635.00
0.00
0.00
0.00

Figure 11-4 Timed Acquisition Setup Full Spectrum Mode

Multiple spectrum files are saved using a data series name, followed by an incremented number, followed by the .SIS extension. Example: Data Series #####.SIS The data series name is entered in the **Data Series** control (See TEST in Figure 11-4) The **Start at #** control selects the beginning number in the series.

The **Select Folder** button opens a dialog box (Figure 11.5) to choose the folder where the spectra will be saved. Once a folder is selected, click on **Select Cur Dir** button to return back to the set-up box.

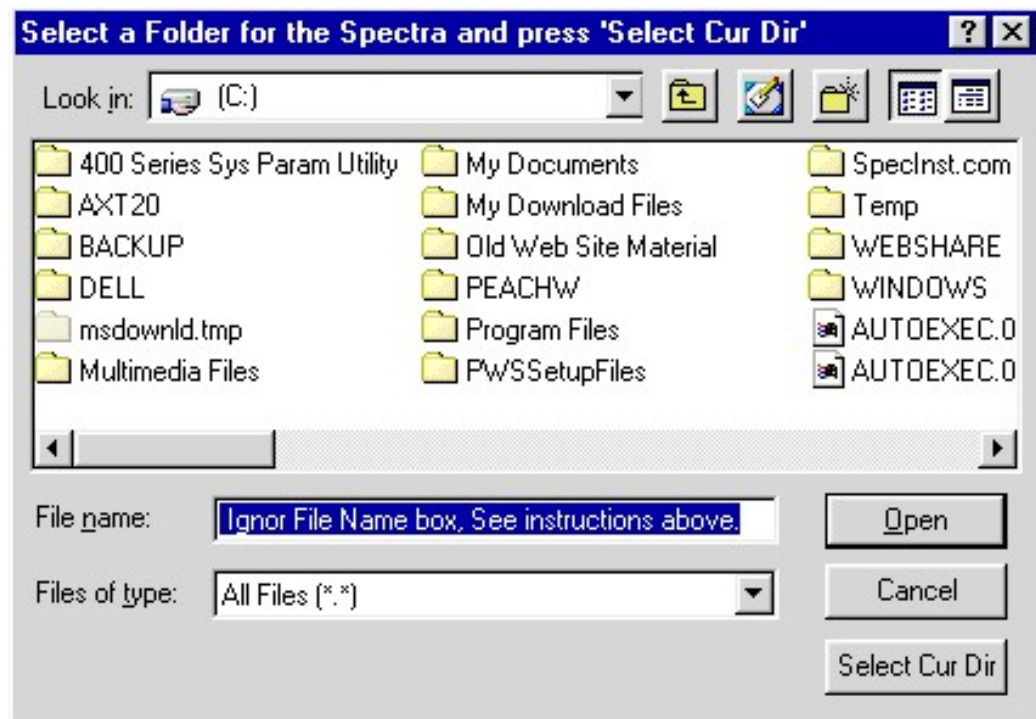


Figure 11-5 Set path dialog box

In the discreet wavelength mode, the parameters to be chosen are the time interval between acquisitions, number of acquisitions, and the wavelengths to be acquired. Up to ten wavelengths can be chosen. Any wavelength set to zero will be ignored.

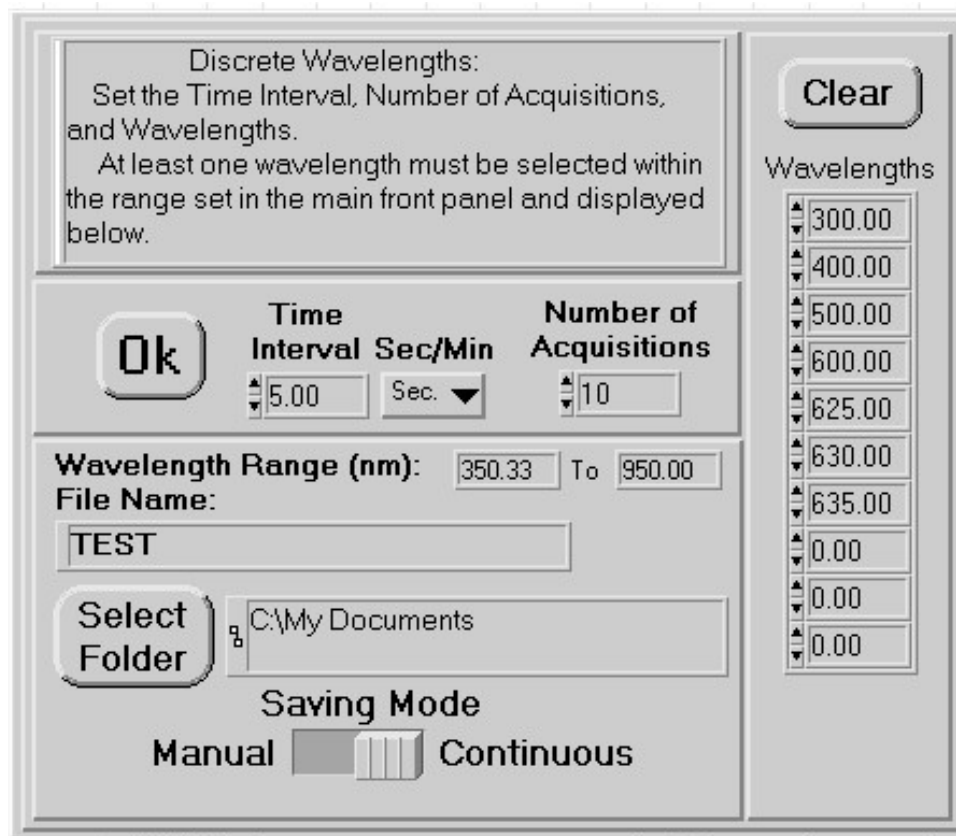


Figure 11-6 Timed Acquisition Setup Discrete Wavelengths Mode

In this mode, only the values at the chosen wavelengths are saved. If the Saving Mode is set to Manual, no Select Folder button is present, as the data for the discrete wavelength acquisition will be saved manually at the end of the run with the Save to File button in the Timed Acquisition panel. When set to Continuous as in Figure 11-6, the Select Folder button is present and the file path is set in a dialog box seen in Figure 11-5. In this mode, the data is saved to the same file after each spectrum is taken. This is useful when doing long time acquisition so the data is not lost if there is a computer problem or power failure. Files saved as discrete can only be opened in the Time Acquisition window

Start - Pressing this button starts acquisition for either full spectrum or discrete wavelength mode.

Time Remaining - This is an indicator that shows the amount of time remaining to complete the data acquisition.

Time Until Next Acq. - This indicator displays the time remaining until the next acquisition will take place.

12. The SI400 Peak Find Front Panel

The Peak Find front panel is opened by pressing the button on the Main front panel and is used to analyze the peaks and valleys of a spectrum. These values are also displayed in a table. A spectrum must have been taken or opened in the Main front panel before analysis in the Peak Find window.

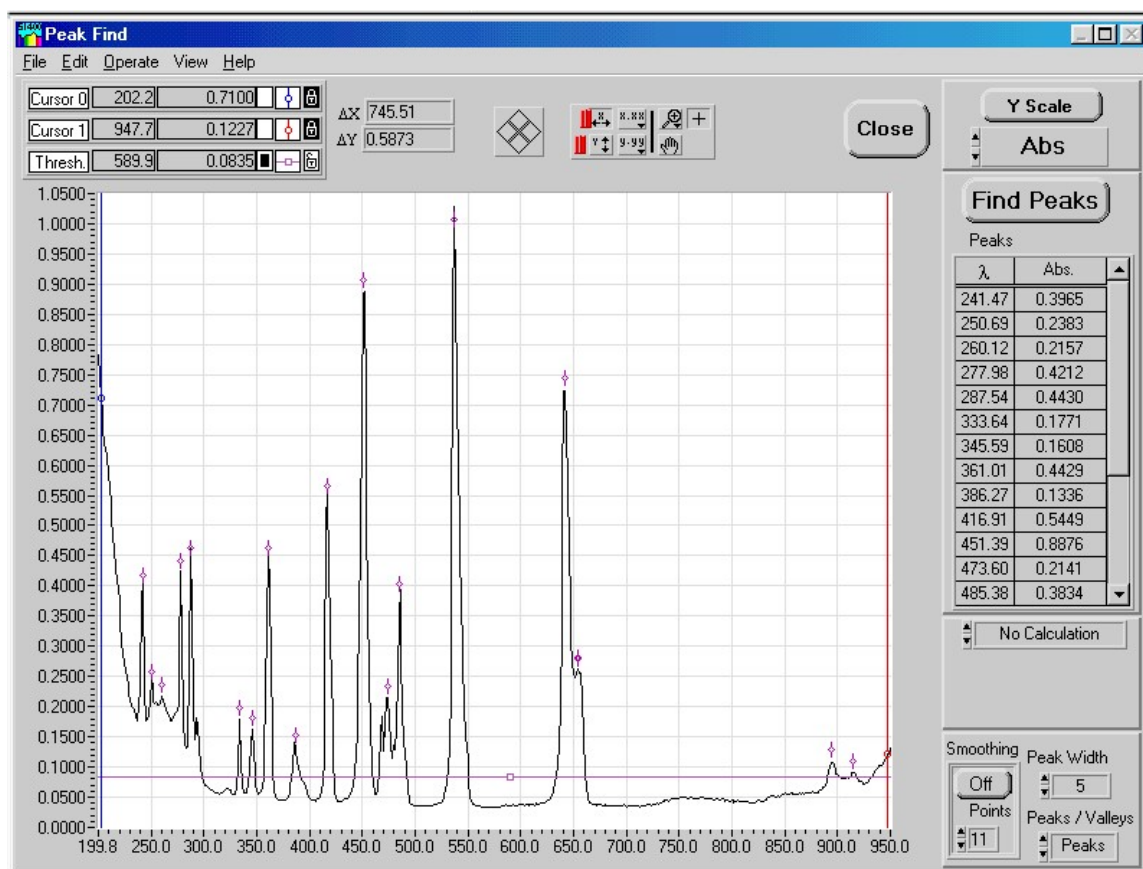


Figure 12-1 Peak Find front panel

12.1 Graphical Display

The graphical display on the Peak Find front panel is used to present absorbance, transmission, reflectance or sample or blank intensity as a function of wavelength. The controls to manipulate the display are discussed in section 7.2.

When the window is opened, the default representation will show the peaks of the spectrum. To view the valleys of the spectrum, select the box in the lower right corner, and choose the valley option.

1.2 Peak Find Front Panel Controls

The following is an alphabetical listing of Peak Find front panel controls.

Close – Closes the Peak Find front panel and re-opens the Main front panel

Film Thickness – Calculates the thickness of substrate in micrometers (um). The user must type in the refractive index of film being tested. This calculation is found under the peaks find table in the “No Calculation” menu.

Find Peaks – Calculates the highest point in each peak, or lowest point in a valley, marks the spectrum and updates table up to 60 points.

Function Key F5

Peak Width – Used in the calculation of determining the peaks.

Peaks / Valleys - Toggle switch to change the display of peaks to valleys.

Print – Prints spectrum and table.

Restore Full View – Displays original spectrum to full scale. This operation is found in the View Menu.

Smoothing - Smoothing - Smooths the data that is displayed by a Savitzky-Golay quadratic-cubic algorithm with an adjustable number of points. This effectively increases the signal to noise ratio of spectral data at the expense of reduced spectral resolution. Data saved to disk files is not saved in smoothed form, but any file read from disk can be smoothed. The smoothing algorithm is described in J. Anal. Chem., 1964, 36, p. 1627.

Points - Varies the number of points in the smoothing algorithm. As the number of points is changed, the current spectrum displayed is dynamically updated with the new smoothing parameters.

On/Off - Turns smoothing on or off. Only the currently displayed spectrum is affected. Once smoothing has been turned on any subsequently acquired spectra, or data files read from disk will be automatically smoothed.

Thresh – The threshold cursor allows the manipulation of the peak find function to only show the peaks above the **thresh** cursor level. In valley analysis, only the troughs below the **thresh** cursor are displayed. The cursor movement is performed the same as the other two cursors.

Y Scale – Adjusts the y-axis to fit the spectrum to fill the full display.

13. The SI 400 Multi Spectra Display

The Multi Spectra Display front panel is opened through the view menu on the Main front panel and is used to see and manipulate up to 10 spectra at a time. To be displayed in this window, the spectra must have been acquired in the Main front panel or as saved disk files. The graphical display is similar to the one on the Main front panel.

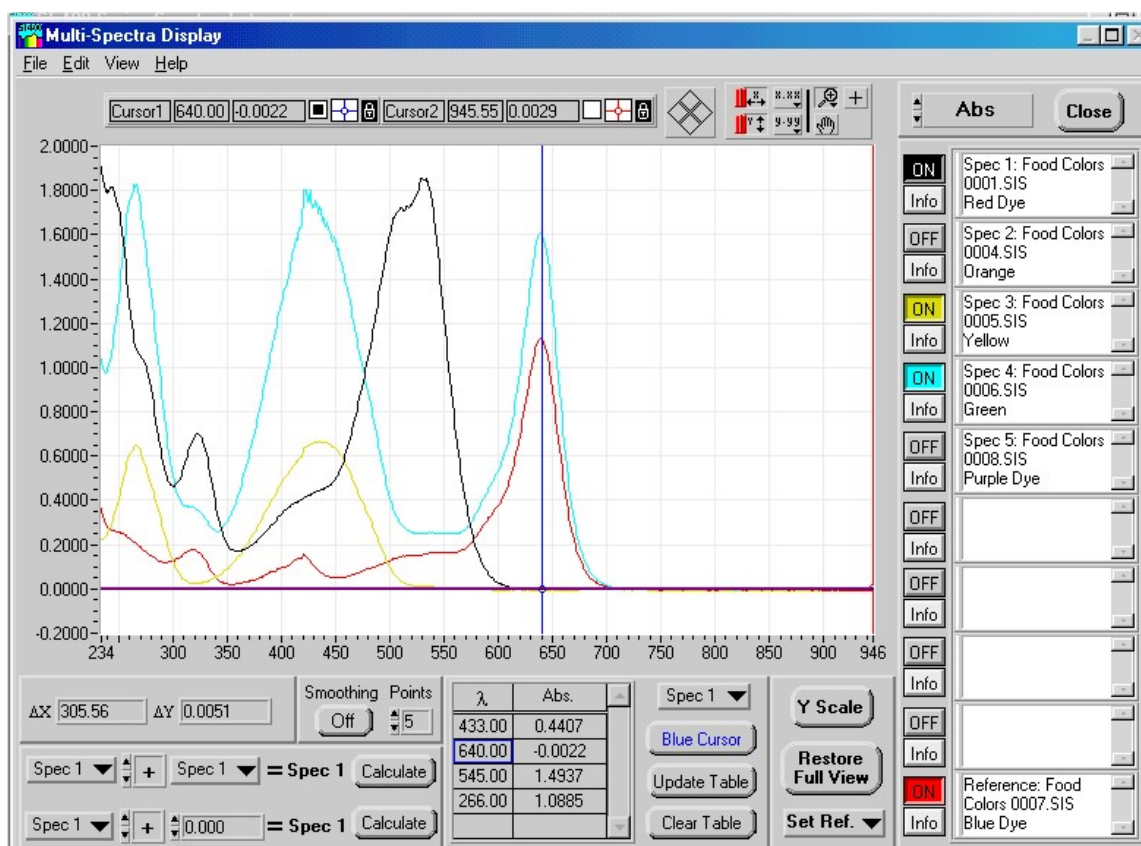


Figure 13-1 Muli Spectra Display front panel

The spectra can be manually opened in the Multi Spectra Display window from saved files or they can be automatically transferred over by selecting the **Auto Add to Multiple Spectra** option in the Main front panel. This feature is on by default. As each spectrum is acquired and transferred to the Multi Spectra Display panel, the spectrum shift down the list. When more than 9 spectrum are opened or transferred, the spectrum are removed from the bottom. The tenth spectrum in the table is a reference cell and will not automatically be removed.

The spectrum in the Multi Spectra Display front panel can be manipulated in simple mathematic functions such as adding, subtracting and multiplying. The arithmetic can be performed on the spectra with two spectra or a spectrum and a

constant. The newly formed spectrum will transfer up to the number one spectrum cell in the table.

13.1 Graphical Display

The graphical display on the Multi Spectra Display front panel is used to present absorbance, transmission, reflectance or sample or blank intensity as a function of wavelength. The controls to manipulate the display are discussed in section 7.2.

The spectra displayed can be turned on and off by selecting the **on/off** button next to each spectrum.

13.2 Multi-Spectra Display

The following is an alphabetical listing of Multi Spectra Display controls

Blue Cursor - Adds an entry into the wavelength column of the data table at the current position of the blue cursor.

Calculate – Performs the mathematical operation between the two selected spectra or spectra and a constant value.

Clear Spectrum – Clears all or individual spectra from the spectra list.

Clear Table - Clears the data table.

Close – Closes the Multi Spectra Display panel and re-opens the Main front panel.

Export Files - From the File menu exports a single file or a group of files that have been saved on the disk to an Excel spreadsheet file or a delimited ASCII text spreadsheet. For instructions, refer to section 9.2.

File Name Filter - limits the sort criteria when retrieving files from the disk.

Grid – From the **View** menu allows user to add or remove the grid lines in the spectra display.

Info – Opens a new window to edit and review File Name and Comment section

Comment	File Name
Yellow	Yellow 2.sis

Sample	Yellow Food Dye
Solvent	Water
Conc.	
Info. 1	
Info. 2	
Info. 3	
Info. 4	

Operator: CGJ

Fri, Jan 21, 2000 Time: 10:44:20 AM Blank Taken: Fri, Jan 21, 2000 at 10:42:03

Figure 13-2 Spectra Info Box

ON/OFF – Adds or removes the spectra from the display window

Open File - Opens a data file created with the **Save File** menu item. The spectrum in the data file, along with all other file information, is displayed.

Open Multiple Files -Opens multiple data files saved on disk. The spectrum in the data file, along with all other file information, is displayed.

Select a Folder – Opens new window to select a folder from which to open files. Click on **Select Cur Dir** to finish operation and return to Open Multiple Files window.

Select Files – Click on individual files or use the shift key for multiple selections up to 9 spectra. To select a contiguous series of file names, shift + click and drag. Click **OK** to finish.

Print Graph - Outputs spectrum only with legend

Restore Full View – Rescales the image to the original settings.

Save – Select the spectrum to be saved and edit comment section in the comment window. Click on **Close** and a save file window opens. Type in file name and click on **Save**.

Set Reference – Selects which spectra in the table is to be used as a reference and transfers all data of the chosen spectra to the reference spectrum

destination. To transfer a spectrum into the reference cell, the spectrum must be turned **ON** to come up in selection choices.

Smoothing - Smooths the data that is displayed and that used for quantitative data analysis by a Savitzky-Golay quadratic-cubic algorithm with an adjustable number of points. This effectively increases the signal to noise ratio of spectral data at the expense of reduced spectral resolution. Data saved to disk files is not saved in smoothed form, but any file read from disk can be smoothed. The smoothing algorithm is described in J. Anal. Chem., 1964, 36, p. 1627.

On/Off - Turns smoothing on or off. Only the currently displayed spectrum is affected. Once smoothing has been turned on any subsequently acquired spectra, or data files read from disk will be automatically smoothed.

Points - Varies the number of points in the smoothing algorithm. As the number of points is changed, the current spectrum displayed is dynamically updated with the new smoothing parameters.

Update Table - Updates the absorbance, % transmission, % reflectance or intensity fields in the data table. Used when wavelengths are manually entered in the data table rather than using the blue cursor/red cursor buttons.

View All – From the view menu, turns all spectra in the table to the ON position, displaying all spectra.

Y Scale – Adjusts the y-axis to fit the spectrum to fill the full display

14. Lamp Adjustments

The tungsten source used in the 400 series instruments is a 10 watt quartz-halogen lamp with approximately 3100°K filament temperature. Its average life expectancy is over 200 hours (longer life, lower output lamps are available; call S.I. Photonics for information). The deuterium source is a high-intensity, pre-aligned lamp with an average life of 4,000 hours. Both lamps need to be periodically replaced as normal instrument maintenance. The tungsten source is replaced only when it fails. The deuterium source is replaced whenever the estimated operating hours exceeds its lifetime, or when the lamp intensity and stability are unacceptable.

Use care in replacing both tungsten and deuterium lamps as the lamp envelopes are fragile and can break during handling. Also, both lamps and their lamp housings become extremely hot during operation. Allow them to cool before disassembling. Finally, the deuterium lamp outputs ultraviolet radiation that is harmful to eyes and skin. Observe the precautions on the replacement lamp, and protect the eyes with **plastic** safety glasses designed to absorb UV light if it is necessary to view the lamp during operation.

14.1 Adjusting Lamp Intensities

Any time the tungsten or deuterium lamps are replaced or adjusted, or the fiber optic probe is changed on the spectrophotometer, it may be necessary to adjust the lamp intensities such that the minimum detector integration time is used, and the CCD detector does not become overexposed. This is accomplished by lamp intensity adjustment screws that prevent a portion of the light from the lamps from reaching the fiber optic probe input.

During all lamp intensity tests and adjustments, the fiber optic probe used with the spectrophotometer should be installed and sampling a "blank". If a solution dip probe is used, it should be immersed in a blank solution. If a reflectance probe is being used, it should be pointed towards a white reflectance standard. The reflectance probe rarely needs the light reduced.

To adjust the lamp intensity, the AC power must be disconnected and the spectrophotometer cover must be removed (see Section 5.2 Removing the Cover). Next, the main power must be reconnected while the cover is removed. **Care must be taken to not touch any electrical components under or near the guards on the main power supply or the deuterium lamp power supply as high voltages exist in these regions.** Also, avoid dropping tools or other metal objects that could cause electrical shorts in the spectrophotometer electronics.

Switch on the spectrometer power and initialize. There is a lamp adjust utility that is accessed from the Configure screen from the Main panel Operate menu. Once the spectrometer is initialized, two additional buttons on the Configure screen become active for adjusting the deuterium and tungsten lamps. When pressed, a lamp adjustment window appears (see Figure 14-1) with a bar scale that monitors the intensity of the particular lamp. The bar is blue when the intensity is too low, green when it is in an acceptable range, and red when the intensity is too high. Locate the appropriate lamp intensity adjustment screw (see Figure 14-2 and Figure 14-3) and adjust the lamp so that the bar is closest to the top of the bar scale while it is still green. At this point, the lamp intensity should be adjusted such that the detector is never over ranged, yet a minimum acquisition time is required.



Figure 14-1 Lamp Adjustment Window

The same Allen wrench used to lock the fiber optic probe ferrules in the spectrometer is used for the intensity adjustment screw (see Figure 14-2). The adjustment screw is self-locking, and is somewhat more difficult to turn than the probe setscrews. Turn the screw in or out to adjust the intensity.

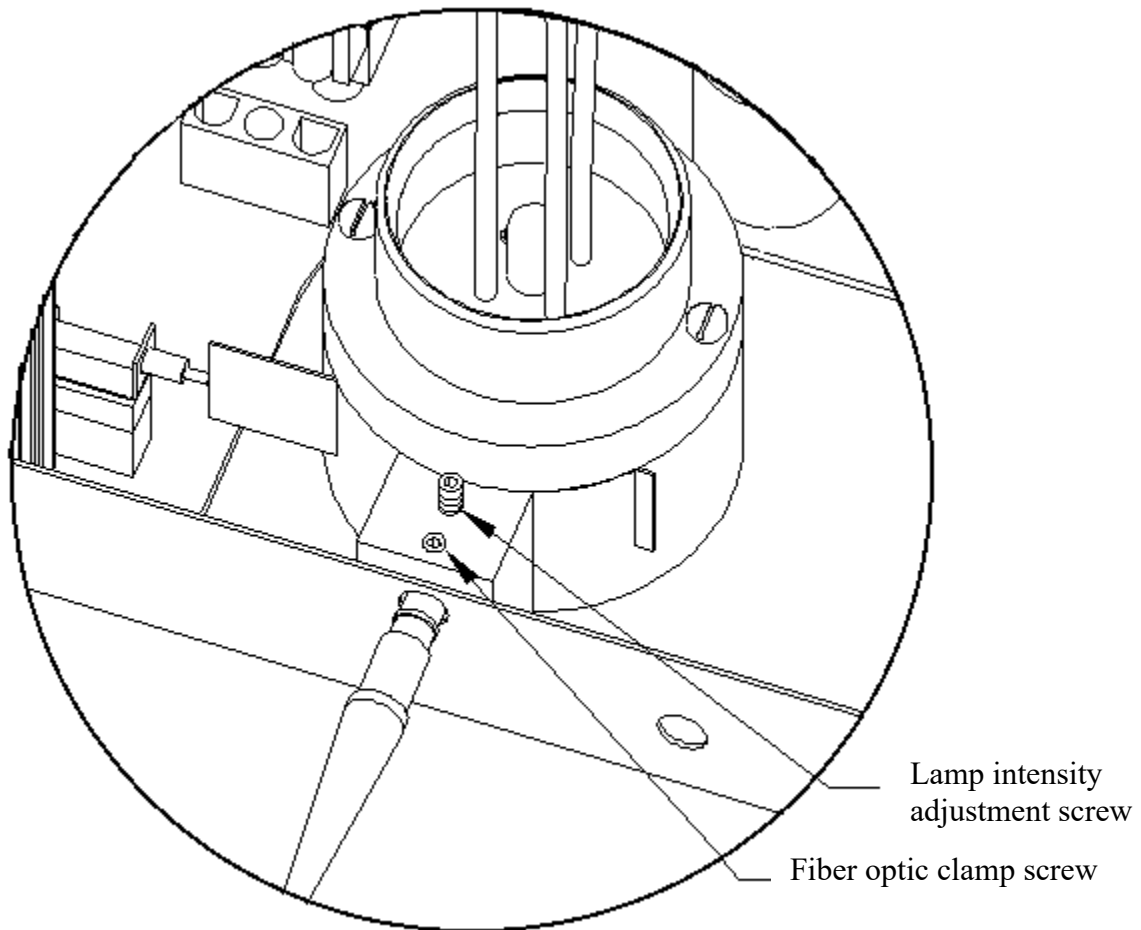


Figure 14-1 Deuterium lamp intensity adjust

14.2 Tungsten lamp intensity

The tungsten lamp intensity is adjusted by rotating a flag into the path of the lamp output (see Figure 14-3). The exposure is very sensitive to the flag rotation. The hex head adjustment screw will rotate the flag; a correct size hex wrench was supplied with the instrument. The flag should have enough rotating resistance to stay in place.

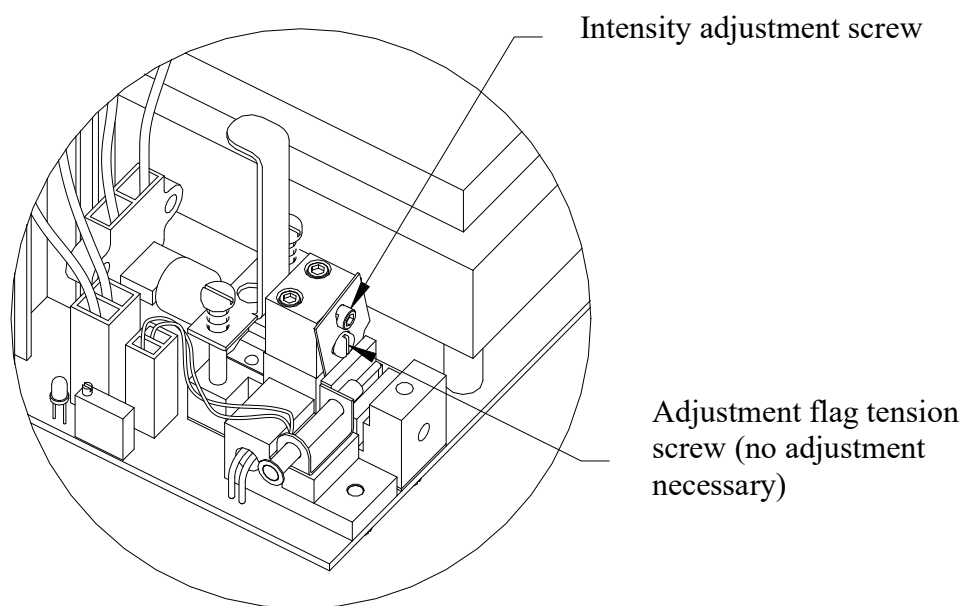


Figure 14-2 Tungsten Lamp Adjustment Flag and Screw

14.3 Tungsten Lamp Replacement

Touching the replacement lamp envelope with the hands during installation can cause premature failure. Replacement lamps are shipped with a plastic or paper cover over the envelope to protect them from being touched. Do not remove this cover until the lamp is installed in the socket, and ready for installation in the lamp housing. If the lamp envelope is accidentally touched, clean it with absorbent lens tissue moistened with isopropyl alcohol.

The AC power must be disconnected and the spectrometer cover must be removed as described in Section 5.1. The tungsten lamp housing on the main power supply circuit board, is located to the left of the main power supply transformer as shown Figure 14-4. The tungsten lamp is retained in the housing with a spring-loaded clamp. Grasp the clamp at the hook in the clamp stem between the two springs and lift straight up to release.

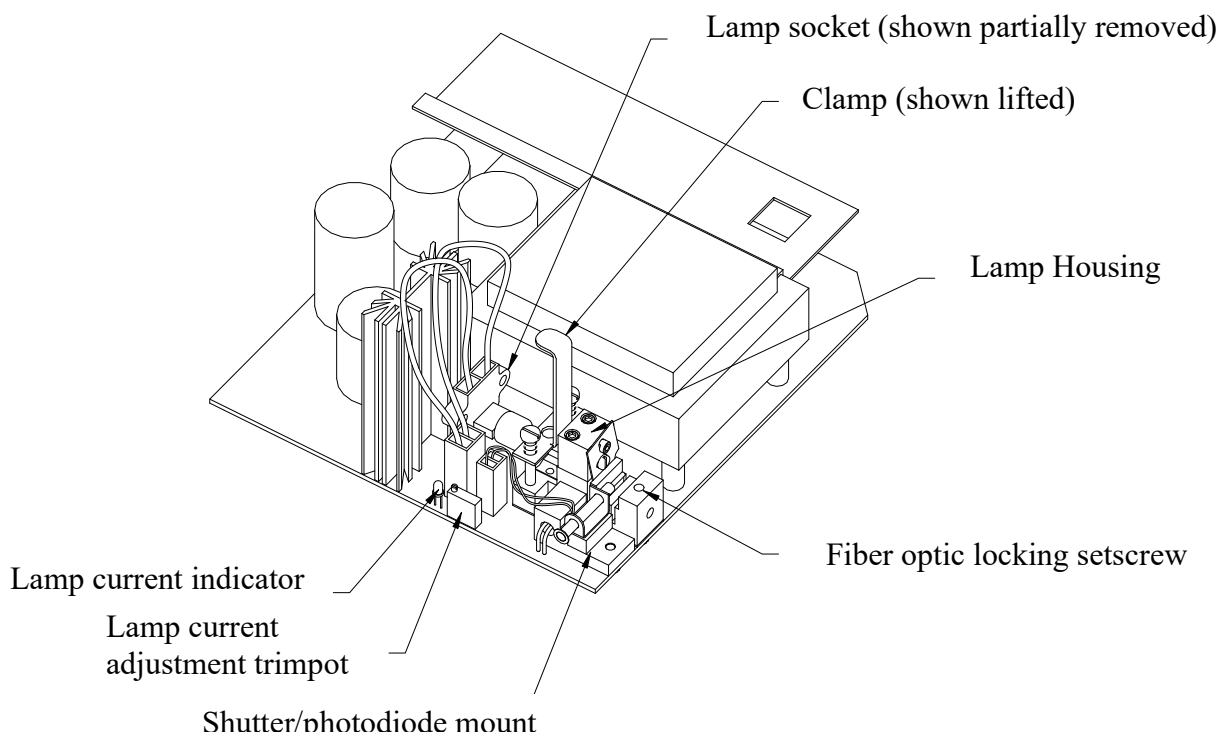


Figure 14-3 Tungsten Lamp Housing Location

Grasp the lamp socket leads and pull the lamp socket from the housing. Pull the lamp from the socket and discard.

Install the replacement lamp in the socket, remove the protective cover, and then set the lamp into place in the housing while again pulling up on the clamp. Before releasing the clamp, make sure that the lamp is properly seated in the housing such that the bulb is not damaged. Gently release the clamp to secure new lamp.

14.4 Adjusting Tungsten Lamp Current

In the event that the tungsten lamp may need adjustment or is removed and re-installed for any reason, the lamp current set point must be adjusted. This is performed by adjusting a trimpot until a LED indicates the proper lamp current. The lamp current is adjusted with the spectrometer cover removed, and power applied. Make sure that the tungsten lamp is illuminated and the deuterium lamp is off before proceeding. Locate the lamp current adjustment trimpot and the lamp current indicator LED (see Figure 14.4). Use a trimpot adjustment tool to turn the trimpot clockwise until the LED is illuminated. Next, turn the trimpot counterclockwise until the LED just becomes fully extinguished. When the lamp current indicator is not illuminated, the lamp current is being regulated at the correct level by an optical feedback circuit.

This trimpot is **NOT** meant for adjusting the lamp intensity. Before placing the unit in service, if necessary adjust the lamp intensity as described in Section 0.

14.5 Deuterium Lamp Replacement

The AC power must be disconnected and the spectrometer cover must be removed as described in Section 5.1. Locate the deuterium lamp electrical connector near the right side of the lamp power supply transformer as shown in Figure 14.5. There is a latch on the right side. Press the top of the latch towards the connector body to release it, then pull straight up on the connector to detach it from the printed circuit board.

The deuterium lamp is held in its housing by two screws located on top of the lamp mount as shown in Figure 14.5. Use a small, flat blade screwdriver to remove these two screws. The lamp can now be lifted from the spectrometer and disposed. Install a new lamp by reversing the procedure above. No optical alignment is necessary.

If necessary before placing the unit in service, adjust the lamp intensity as described in Section 14.1.1.

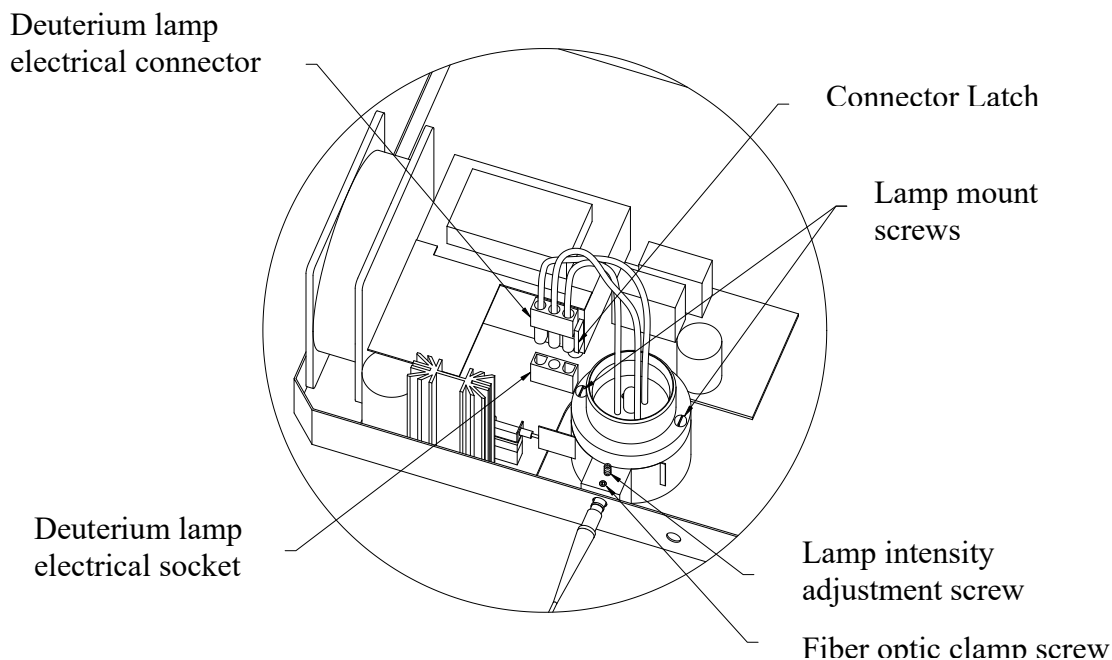


Figure 14-4 Deuterium Lamp Housing and Electrical Connector

14.6 Lamp Warm Up Times

Both tungsten and deuterium lamps require a warm up period of approximately 1 hour before they reach maximum stability, but for routine use, 30 minutes may be sufficient. The lamp warm up time is independent of the time necessary for the spectrograph temperature to stabilize. This is especially important to remember if the deuterium lamp is not turned on when the instrument is first powered up. If either lamp has not had sufficient warm up time, baseline drift will be observed.

15. Specifications

Performance	Model 410	Model 420	Model 430	Model 440
Wavelength Range	350-950 nm	200-950 nm	350-980 nm	190-980 nm
Spectral Bandwidth	1.2 nm	1.2 nm	<1.1 nm	<1.1 nm
Wavelength Accuracy	1.2 nm	1.2 nm	<1.1 nm	<1.1 nm
Photometric Range	0.002-3.00 AU	0.002-3.00 AU	0.002-3.20 AU	0.002-3.20 AU
Photometric Accuracy	0.005 AU	0.005 AU	0.005 AU	0.005 AU
Baseline Stability	<0.005 AU/hour	<0.005 AU/hour	<0.005 AU/hour	<0.005 AU/hour
(at 300 nm and 600 nm)				
Stray Light	<0.02%	<0.02%	<0.02%	<0.02%
(at 300 nm and 600 nm)				
General				
Warm Up Time	½ hour	½ hour	½ hour	½ hour
Dip Probe Pathlength	~1 cm	~1 cm	~1 cm	~1 cm
Cell Holder Beam Diameter	0.20"	0.20"	0.20"	0.20"
Size	9" x 13" x 5.5"	9" x 13" x 5.5"	9" x 13" x 5.5"	9" x 13" x 5.5"
Weight	12 pounds	15 pounds	12 pounds	15 pounds
Power	110/220 VAC 0.8A	110/220 VAC 0.8A	110/220 VAC 0.8A	110/220 VAC 0.8A

16. Troubleshooting

16.1 Spectrophotometer Initialization and Associate Error Messages

If initialization is not successful, one of three error messages will appear in dialog boxes. The first possible error message is:

Error 37 occurred In Com Port Init.VI:

The selected COM port either does not exist or is in use by another application. Press the CONFIGURE button to select another COM port. Refer to user's manual for more information.

Possible reasons:

LabVIEW: Device not found.

The SI400 control application could not find the COM port you specified during configuration. The COM port may physically exist or not, but it could not be "found" through the Windows operating system. If another Windows application is open which may be using the COM port, close the application. If you are sure that the COM port physically exists, make sure that Windows knows of its presence. A good place to start troubleshooting this problem is for Windows 95 and higher, open the System application in the Control Panel and look under Device Manager, Ports, for the COM ports that are available.

The second error message that can occur is:

Error -1201 Occurred

Spectrometer Initialization.

Series 400 spectrophotometer is not responding to initialization command. Verify that spectrophotometer power is switched on, and that the communications cable is connected.

This will arise if the communications port is found on the host computer end, but there is no response from the 400 Series Spectrophotometer when information is sent to it. Make sure that the RS-232 cable is plugged into the spectrometer, and to the port that you specified during configuration. Also, make sure that the communications cable has no broken conductors. The 9 pin D male to 9 pin D female connector cable supplied with the 400 series instruments is wired straight through with pin 1 connecting to pin 1, pin 2 to pin 2, etc.

A third message that can appear is:

Error -1202 Occurred

Spectrometer Init.VI.

An error has occurred while transmitting the initialization file to the series 400 spectrometer. A likely cause is a mismatch in the communications data rate (baud rate) settings. The computer port baud rate must match the spectrometer baud rate. The computer baud rate is changed using the CONFIGURE button on the main program screen. Refer to user's manual for information about the spectrometer baud rate.

If the COM port baud rate is correct, check the integrity of the communications cable.

This error indicates that while there was a response from the spectrophotometer, it was not the expected one. In addition to checking baud rates, make sure that the COM port selection is correct. If the SI400 application is trying to communicate with the spectrophotometer on a serial port that another device (such as a mouse or modem) is connected, this error can result. It is also necessary that the COM port being used is not sharing an interrupt (IRQ) with another COM port or modem.

This error can also occur if the cable between the host computer and the spectrophotometer is too long, or is being used in an electrically noisy environment, or is not properly shielded. If it is necessary to use a long cable, try to reduce the baud rate to 9,600.

Sometimes it is necessary to set the serial transmit buffer to Low in Windows 95. This is performed in the **System Properties** window launched from the **Control Panel**. Select the **Device Manager** tab; expand the **Ports (COM & LPT)** item; double click on the COM port being used to display its properties. Next select the **Port Settings** tab and then press the **Advanced** button to open the **Advanced Port Settings** window. Here there are two controls, one for Receive Buffer and one for Transmit Buffer. The Receive Buffer is normally set near High and should be left this way. The Transmit buffer needs to be set to **Low** for the most reliable communications.

16.2 Unreliable Communications

Unreliable communications can result from a number of sources, including the computers RS-232 port, cable, and software drivers. One of the primary sources of problems is slow computer response time that is related to how many tasks the Windows task scheduler has in its queue. If the system can not respond to the serial communications port sufficiently fast, characters may be occasionally

and unpredictably dropped, resulting in errors during the spectrophotometer initialization.

The use of a buffered serial interface port equipped with 16550 UARTs is required, as it will collect characters internally while waiting for service from the computer's processor. The older 8250 type UARTs has no internal buffer, and will not work reliably. The type of UART used in a serial port can be determined using the DOS 6.22 "MSD" command.

The use of 16550 UART equipped serial ports and high quality RS-232 cables will result in reliable communications in nearly every situation. If communications problems persist, contact Spectral Instrument's service department for assistance with other solutions.

It has been found that on some notebook computers that it is necessary to turn off the power management to obtain reliable communications. This is due to the overhead added to the system by the power management causing some communications to be lost.

16.3 Screen Size

The programs front panels are designed for use with at least 800 x 600 resolution on the Windows display. If the display is set to 640 x 480, parts of the front panel will be off the screen and it is necessary to change the resolution of the display. See the Windows manual or Help screens for the procedure to change the display resolution.

16.4 Lamp Intensity Warning Messages

Figure 14-1 shows what a normal Blank intensity spectrum looks like. This spectrum will vary from one spectrometer to another and depending on where the crossover wavelength for the UV and visible lamps is set. Here the crossover is at 460 nm and is not optimized.

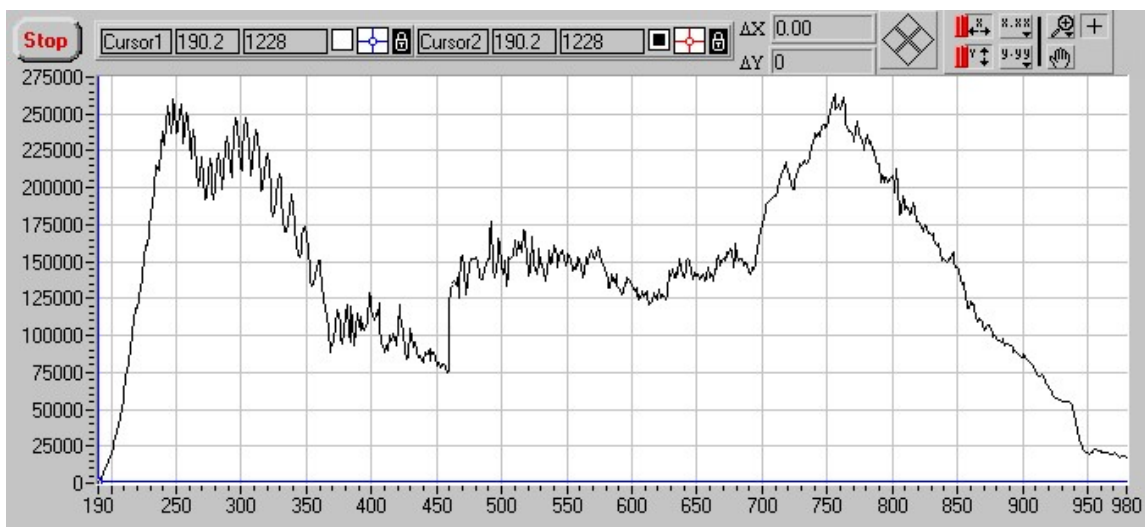


Figure 0-1 Normal Blank intensity spectrum

16.6 High Lamp Intensity

The SI400 application will alert the user if the tungsten lamp intensity is too high when a blank is acquired by giving the warning message:

The Tungsten source intensity is too HIGH and must be adjusted. See the manual for instructions.

A similar message will result from high deuterium lamp intensity. The user may choose to either cancel the operation or continue by pressing the appropriate buttons in the dialog box.

If either warning message occurs, turning the intensity adjustment screw clockwise should reduce the appropriate lamp intensity (see Section 14.1).

16.7 Low Lamp Intensity

If the light intensity reaching the detector from the tungsten lamp is very low, the warning message:

The Tungsten source intensity is too LOW and may need to be adjusted or the lamp is out. See the manual for instructions.

will appear. A similar message results from a low deuterium lamp intensity. Again, the user may cancel or continue by pressing the appropriate button.

A low intensity means that the spectrophotometer has determined that even by increasing the readout integration time to a maximum (10 seconds for both tungsten and deuterium sources using "normal" precision), the intensity of light reaching the detector is so low that an unacceptable signal to noise may result.

Light may not reach the detector due to:

- 1) A lamp is not functioning.
- 2) The fiber optic probe is not installed, not sampling a blank, or very inefficient or defective.
- 3) Lamp intensity adjustment screw is blocking too much light.

Check each item above in the order listed. Adjust the lamp intensity as described in Section 14.1.

17 Index

A

[Acquiring Absorption Spectra · 24](#)
[Auto Save · 35](#)
[autoscaling · 19](#)

B

[Baud Rate Selection · 12](#)

C

[COM Ports · 21](#)
[Configure · 21](#)

D

[deuterium · See lamps](#)

E

[Errors](#)

Error -1201 · 65
Error -1202 · 66
Error 37 · 65

[Export Files · 30](#)

[External Lamp Connector · 26](#)

[External Shutter Connector · 25](#)

F

[Fiber Optic Sampling Accessories](#)

Cuvette Cell Holder · 24
Dip Probe · 23
Installation · 10
Reflectance Probe · 24

[Function Keys · 20](#)

G

[Graphical Displays and Associated Controls · 18](#)

I

[Integrating Sphere Accessory · 26](#)
[Initializing Spectrophotometer · 22](#)

K

[kinetics · 46](#)

L

[Lamps](#)

Adjusting Lamp Intensities · 57
Adjusting Tungsten Lamp Current · 60
Deuterium lamp intensity · 58
Deuterium Lamp Replacement · 61
Tungsten lamp intensity · 59
Tungsten Lamp Replacement · 59

[linear regression · 42](#)

M

[Mult Spectra Display · 53](#)

P

[Peak Find · 51](#)

[Printing · 34](#)

Q

[Quantitative Analysis · 39](#)

Quantitative Wavelengths Selection ·
44

R

RS-232 Communications · 11

S

Save As Excel or Text file · 35

Save Setup · 36

Save to File · 35

Smoothing · 36

Software Installation · 15

Specifications · 63

T

Technical Assistance · 5

Timed Acquisition · 44

Troubleshooting · 65

tungsten · See lamps

V

Voltage Selection · 10

W

Wavelength Range · 37

Z

zoom controls · 19

