Applied NanoFluorescence

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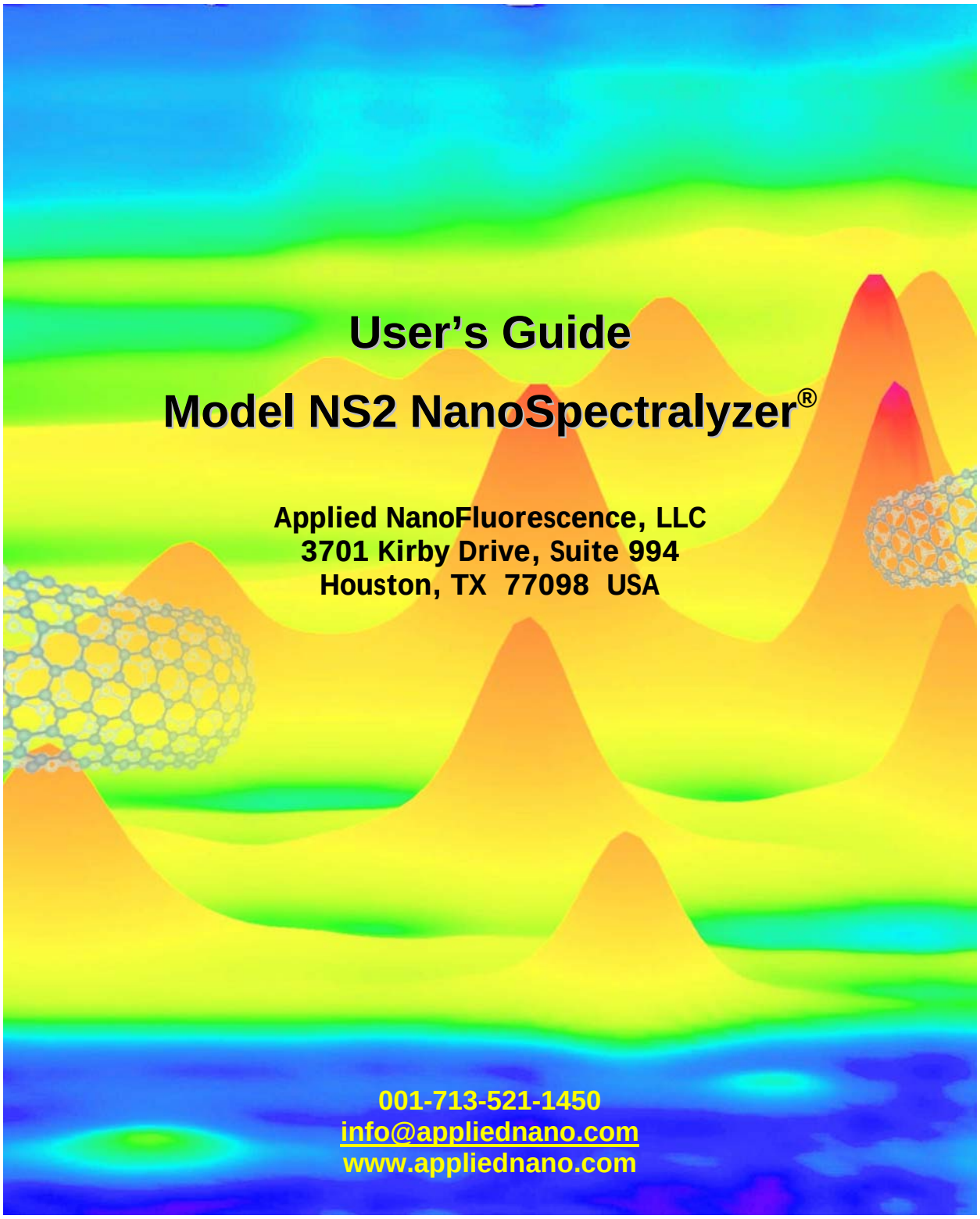

User's Guide

Model NS2 NanoSpectralyzer®

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Applied NanoFluorescence

Contents

1. Introduction

The model NS2 NanoSpectralyzer® from Applied NanoFluorescence, LLC (ANF) is a unique, state-of-the-art scientific instrument (the Instrument) designed specifically to characterize samples of carbon nanotubes using absorption, near-infrared emission and Raman spectroscopy.

The Instrument is optimized for high sensitivity and small sample volumes. It can also measure data in rapid sequence for concurrent kinetic detection of fluorescence, absorption, and Raman spectra.

The NS2 NanoSpectralyzer will let you perform quick, non-destructive detection and analyses of single-walled carbon nanotubes (SWCNTs) in aqueous or other suspensions to determine diameter distributions, (*n*,*m*)-level compositions, sample condition, and to monitor time-dependent changes in these properties.

2. Installation

The Instrument consists of a Main Optical Module with a hinged sample door on its top panel, a NIR Spectrometer Module containing a near-infrared spectrometer and 512-element InGaAs detector array, a Raman Spectrometer Module, a Raman Laser Driver, and a computer system with pre-installed MS Windows 7 Pro, Origin software (OriginLab), and **NanoSpectralyze**® software (ANF). **NanoSpectralyze**® (the Software) is the custom program used for Instrument control, data acquisition, data interpretation, and results display (including creating Origin project files).

Unpack the NS2 system from shipping containers and check carefully for signs of damage in shipment. Immediately notify the shipper and ANF if any damage is found. The following items are usually included (check against packing lists for more specific configuration):

- Main Optical Module
- 12 VDC external power adapter for Main Optical Module, with round multi-pin connector and line cord
- Cable to connect Main Optical Module (9-pin D-sub connector) to computer USB port (includes serial-to-USB adapter)
- NIR Spectrometer Module (in foam-lined plastic case) with attached fiber optic cable and external 12 VDC power adapter
- Cable to connect NIR Spectrometer Module (miniature round multi-pin connector) to a standard USB computer port
- Raman Spectrometer Module with orange fiber optic cable and USB cable
- Raman Laser Driver Module, with power cable
- A PC computer (including a wireless keyboard and mouse) with preinstalled Windows 7 Professional, Origin, and NanoSpectralyze® software
- Surge suppressor power strips
- Two 19" LCD monitors with DVI cables
- Two 10 x 10 mm and two 10 x 4 mm sample cells (Starna Cells, Inc. parts 1-Q-10GL14-C and 9-Q-10-GL14-C)

- Film sample adapter (fits samples 10 to 12 mm wide by 5 to 45 mm high)
- Temperature control (water-jacketed) sample cell (Starna Cells, Inc. part 62-Q-10)
- Solid fluorescence reference sample
- Sample cell shim plate and magnetic pick-up tool

Prepare suitable laboratory space for locating the NS2. The modules are identified in Fig. 1 below. The footprint of the Main Optical Module is approximately 320 mm wide. You will also need nearby space for the two Spectrometer Modules, Raman laser drive, computer, monitors, keyboard, and mouse. A typical installation, shown in Fig. 2, is approximately 1200 mm wide including dual computer monitors. The Instrument is not very sensitive to normal laboratory vibrations and should function well when placed on an ordinary solid desk, a lab bench, or an optical table. However, as with any precision measurement instrument, extreme and continuous vibration must be avoided. The Main Optical Module is linked to the Spectrometers by optical fibers. Be sure to keep these fiber cables in positions where they will not be damaged, moved, or disturbed.

Contact ANF or your distributor to schedule on-site installation and user training.

Fig. 1. NS2 modules (control computer is not shown).

Fig. 2. Typical layout of the installed NS2 system.

Fig. 3. Back panels of the NIR Spectrometer module (left) and DPSS Laser Driver module (right). On the DPSS Laser Driver, the high / low output toggle switches should remain in the up (High) position except during alignment or service.

3. Hardware Operation

CAUTION - Use of controls or adjustments or performance of procedures other than those specified herein may result in hazardous laser radiation exposure.

- Check that the surge protector strip used to power the NS2 system is switched on, and boot up the control computer if it was turned off.
- Before starting the Instrument, ensure that all cables are properly and securely connected (if a USB cable connection was changed, see Attachment for more details).
- Set the power rocker switch on the front panel of the Main Optical Module to its ON position. The adjacent green LED glows when the module is powered. The second, amber LED glows when the sample hatch is closed, satisfying the safety interlock. The red LED glows to indicate laser activity.
- Set the power rocker switches on the Raman Laser Driver and the Spectrometer Modules to the ON positions. (see Fig. 3 for switch locations on the NIR Spectrometer and Raman Laser Driver)
- Launch the NS2 control program (NanoSpectralyze) by clicking on its Windows desktop icon, normally labeled ANFSoft.
- If a dialog box quickly appears indicating a failure of connection or power, click "Try again". (This symptom may occasionally appear with fast computers.)
- In normal start-up, a "Nanoscape" window will appear showing your software version and the registered hardware configuration. Click OK to close the start-up information window. The NIR Spectrometer Module fan will activate as cool-down begins, and its current detector temperature will be displayed on the main program screen.
- When the NIR detector temperature stabilizes, a dialog box will appear. It can be closed by clicking OK or with a Space or Enter keystroke.
- In a case of problems, look for help in Section 10, **Troubleshooting**.
- The NS2 is designed to accept sample cells with standard outer dimensions of 12.5 x 12.5 mm. The Instrument's optical axis ("Z-value") is set to 8.5 mm above the pad at the bottom of the cell holder for compatibility with many specialized cells, including flow cells and micro-volume cells that are stocked by commercial suppliers. However, when a sample cell is used that does ***not*** require an 8.5 mm Z-value, a shim plate can be placed into the cell holder to raise the cell and allow use of smaller sample volumes. The shim plate, shown in Fig. 4, is made of ferromagnetic stainless steel (with a rubber pad on top) to allow easy placement and removal with a magnetic pick-up tool (supplied). Fig. 4 illustrates shim removal using the tool. To replace the shim, use the magnetic tool to position it at the bottom of the cell holder (pad side up), and then hold in place with a pencil or wooden rod while raising the magnet.

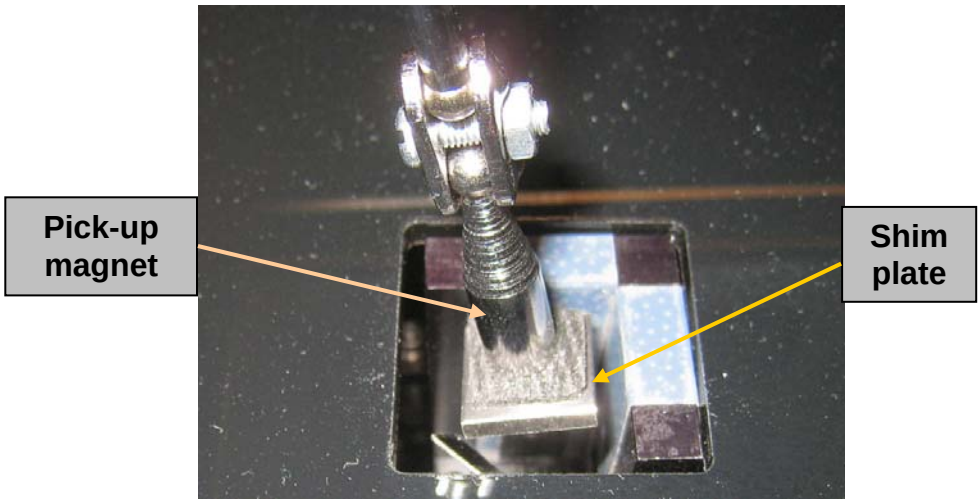


Fig. 4. Insertion of the shim plate into the sample cell holder using the magnetic pick-up tool. Note that the rubber pad faces up and is in contact with the magnet.

- Cells with 10 x 10 mm optical paths and four polished faces allow uninterrupted sequential measurement of fluorescence, absorption, and Raman spectra. With the shim installed, a minimum sample volume of 300 µL is recommended in such cells. Smaller sample volumes (120 µL and 60 µL, respectively) can be used in cells with 10 x 4 mm or 10 x 2 mm optical paths (only two polished faces are required). However, in this case the orientation of the cell must be adjusted according to the type of spectrum being measured. For fluorescence and absorption, the cell's 10 mm path length must be parallel to the long axis of the Main Optical Module. For Raman, the cell must be rotated so that its 10 mm path length is perpendicular to the long axis of the Main Optical Module. These orientation requirements apply both to reference and sample measurements. Orientation marker labels can be found on the bottom of the sample hatch.
- When using the Film Sample Adapter option, place the sample under the spring clip with the region of interest facing the back when viewed in the orientation shown in the Figure 5 photo. Usable dimensions for film sample substrates are 10 to 12 mm wide by 5 to 45 mm high. Note that the adapter must be inserted into the NS2 standard sample cell holder with the face labeled "Top" upward and oriented so that it reads normally when viewed from the front of the instrument. The lower and upper white lines shown on the left post mark the excitation beam heights when the shim plate described above is installed or removed, respectively.
- To avoid optical damage to film samples during measurements, it may be necessary to reduce the incident laser powers below their normal levels. On units with the Film Sample option, such power adjustments are possible for the 638 and 787 nm excitation lasers using the dialog box shown in Fig. 5. This dialog box is accessed in Setup Mode, and any power settings made there will be applied for data acquisitions using the main window. To make a power adjustment, first go to **Setup** mode and set any of the detectors to acquire **Continuously**. Then activate the laser (638 or 787 nm) whose power

is to be adjusted, enter the target power level into the field labeled **Set** in the dialog box, and click on the button to its right. The actual power level, which is displayed in the box labeled **Read,** should then closely approach the target value. It will remain at that level until reset through the dialog box.

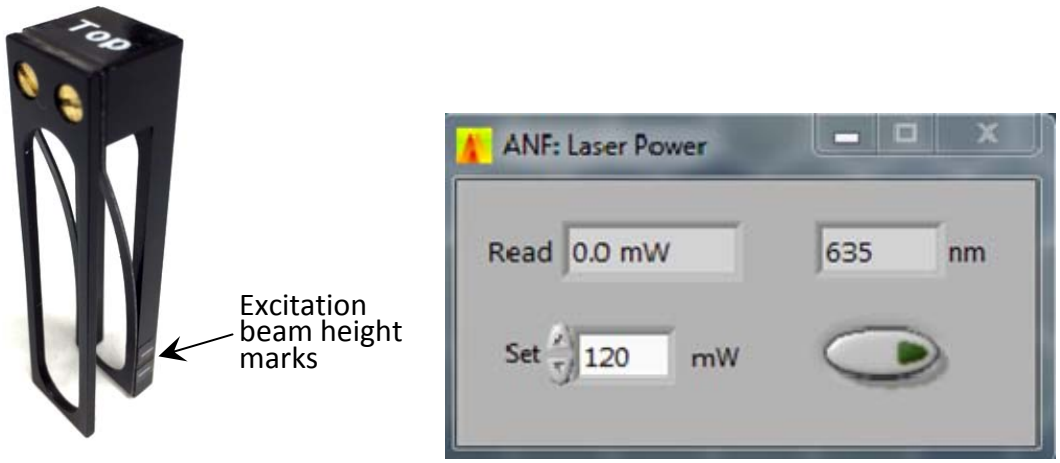


Fig. 5. Film Sample Adapter and Dialog box (available in Setup Mode) for controlling laser output powers.

- Open the sample door on the top of the Main Optical Module. (This door is safety-interlocked to switch off the lasers when opened.) Then place a blank reference cell containing only solvent (normally H_2O or D_2O) and surfactant into the sample holder. To ensure the most consistent amplitude measurements, mark your sample cell so that you can reproducibly orient it with the same face against the front wall of the sample holder. (Note: in many less critical applications, inexpensive disposable plastic cells may be successfully used instead of glass cells. However, ***be cautious*** when using plastic cells with the shim plate, because distortions in the lowest portions of the cell faces may cause baseline errors and reduced signal strengths.)
- Close the sample door. The amber (interlock status) LED should now be lit. Then begin data acquisition by following screen prompts and setting parameters in the NanoSpectralyze program. (These are described in section 5 below.) The red LED will show laser activity during fluorescence or Raman measurements.
- When measuring absorption spectra, it is advisable to wait a few minutes after power-up to allow full probe-light lamp warm-up and stabilization. Also, the highest absorption peaks should not exceed the Instrument's reliability limit of ~3 AU. Dilute the sample with surfactant solution as needed to bring the absorbance peaks within these measurement limits. Finally, note that H_2O absorbs strongly beyond ~1360 nm, so D_2O should be used instead as a solvent if accurate spectral data are required between 1360 and 1600 nm.

- For reliable absorbance results, be sure to use a blank with the same solvent and cell geometry as the sample. Otherwise refractive mismatches may cause systematic errors in the displayed spectra. When using D_2O-based samples, the levels of H_2O contamination in sample and blank cells should be well matched to avoid artifacts at longer wavelengths. To obtain absorptivity results per centimeter of optical path, enter the correct optical path used in the experiment in the Absorption Properties dialog box (see below).
- During a data acquisition run, you will be prompted by a dialog box and an audio message to place the reference (blank) cell or sample cell into the Instrument at the appropriate time. If you wish to change the audio messages, just replace the corresponding .wav files found in the folder C:\ANFSoft\Sounds.
- After completing a data acquisition session, exit the NanoSpectralyze program. This will put the NIR Spectrometer Module into a low-power standby mode and extend the service life of its components. When the software is closed, turn off the power rocker switch on the front panel of the Main Optical Module. After 5 seconds, the other modules will power down automatically. It is usually convenient to leave the control computer powered up, unless it will not be used for a long period.
- When performing long calculations with the software without data acquisition, it is recommended to switch the hardware off. To do this, first go through the normal start-up procedure. Then switch off the Main Optical Module. When your calculations are complete, it will be necessary to switch it back on to properly exit the NanoSpectralyze program. Note that the normal version of NanoSpectralyze software will not run without a connected and powered NIR Spectrometer, which serves as a hardware key even if it is in stand-by mode without probe light and cooling. A special "no hardware" stand-alone version of the software (fully functional with import-export functions and calculations) is also available as an option; contact ANF for details.

4. Sample Preparation

Because SWCNT fluorescence is strongly suppressed by nanotube aggregation, fluorescing SWCNT samples must be prepared using effective dispersion procedures. Each laboratory should develop its own standard protocols appropriate for its range of samples and analytical needs.

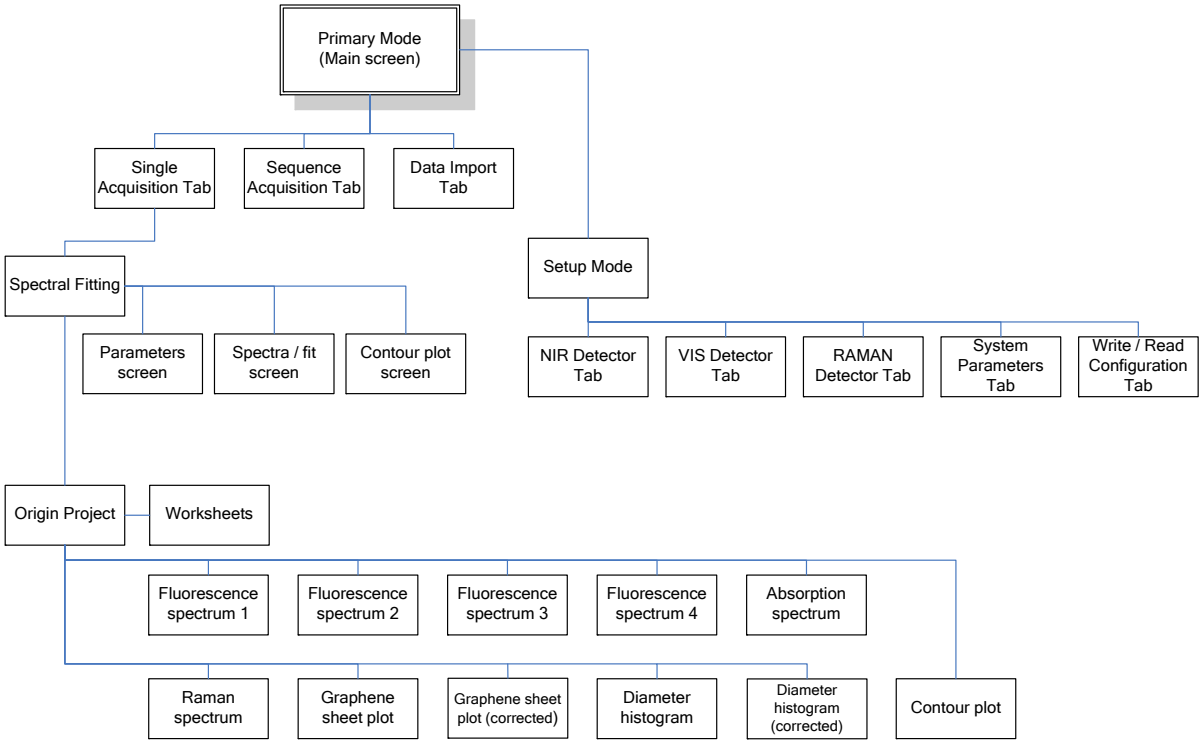
In one recommended sample preparation method, a few micrograms of solid SWCNT is placed in a small glass vial to which are added several mL of a surfactant solution in D_2O. Sodium dodecylsulfate (SDS), sodium dodecylbenzenesulfonate (SDBS), sodium cholate (SC), and sodium deoxycholate (SDOC) are common recommended surfactants. Brief agitation using an immersion-tip ultrasonic liquid processor (such as the Misonix XL2000) should then give a gray homogeneous suspension. For some cationic surfactants (especially SDS), one drop of 1 M NaOH should be added to ensure high and stable fluorescence intensities.

Other published and user-developed sample preparation methods, involving different media, dispersants, agitators, and preparation protocols may also be used.

The NS2 is very sensitive. With high-quality samples, it can record very good fluorescence spectra even from suspensions that appear perfectly clear to the eye. Fluorescent spectra have been acquired at ANF on samples with optical density of only 0.00001 (10^{-5}) at the excitation wavelength. SWCNT-containing films or thin liquid samples with thickness down to a few micrometers, may also be used in the fluorescence measurements (special cells or sample holder adapters may be required).

5. Software Overview

NanoSpectralyze® Software Structure



NanoSpectralyze® is the computer program (the Software) that performs instrument control, data acquisition, data analysis, and results display in the model NS2 NanoSpectralyzer system from Applied NanoFluorescence, LLC. Current versions of the Software are compatible with all commercial NS2 units. The Software consists of executable module(s) and multiple Microsoft Windows and third-party libraries and drivers, and uses specific data in additional files. The Software works in conjunction with the commercial program Origin (from OriginLab Corp.) to generate presentation-quality graphs of the results and to store analysis parameters and templates. A licensed and registered copy of Origin, as well as other software components, has been preinstalled on the computer supplied as part of your NS2 system.

ANF-specific components of the software installation are placed, by default, in the folder **ANFSoft** on system drive C. The folder contains the main executable program, some DLL libraries (in subfolder DLL), files required for detectors (in two subfolders), and sound files (sounds subfolder). The subfolder **Help** contains a brief history of changes and instrument manuals. By default, the main **ANFSoft** folder also contains Origin plot templates (**otp** files), which should not be removed but may be modified according to user needs and preferences. Also present are Calculation templates (Origin **opj** project files) and **Global Template**

Default.opj, which is accessed during Software startup. Additionally, there are two other files, **Sim3D.otm** and **ProfilSWCNT.ogs**, which are used by Origin after fitting to create an output file. Files of type **3DT** are binary files containing a style of 3D plot used by the Software during and after sequence acquisition. The file **style.3DT** is required; the others can be saved and used as desired. Usually, a subfolder named Install contains all third-party installations (excluding Origin) necessary to reinstall the system. In the case of a serious computer problem, a complete backup copy of the folder **ANFSoft** will help to restore the Software. The backup can be also used to reinstall required components if the computer is replaced. Note that a standard way to recover Windows with all software intact is to restore from a previously saved complete backup of the system drive with System State to a new Windows computer.

Administrator access rights under Windows are not required to use the Instrument and the Software. Moreover, it is always preferable to use the PC in a normal "user" or "advanced user" account. However, because of current Windows security features, **administrator** access will be required if any cable connections to the computer are changed, or if a USB port is used that never before handled the external device. (See the Troubleshooting section for more details)

NanoSpectralyze is designed to allow simple, turn-key operation by operators with limited laboratory expertise. However, more advanced and non-standard measurements are also supported. The program has two operating modes. The primary mode is entered by default when the Software launches. It controls automated sequences that perform spectral measurements, analyze the data, and prepare publication-quality graphs of the results. The secondary mode is reached by selecting the "**Setup**" button at the bottom of the main screen. This secondary mode allows real-time display of emission and absorption spectra with full manual control of light sources and data acquisition parameters. The Setup mode does not perform data analysis, but it can be very useful for quickly comparing samples, checking their suitability for detailed measurements, and estimating suitable parameters for primary mode data acquisition. It can be also used for some non-standard experiments that might not be available in the automatic main mode (such as recording fluorescence excited by simultaneous excitation with multiple lasers). Setup mode is also used to change some system parameters and for other service functions.

6. Software Controls and Indicators

Controls and indicators are labeled above or to the right. Some controls share labels with other nearby controls. A pop-up box with information or a usage tip may appear when a mouse cursor is placed above certain controls. Some controls and indicators can be unavailable depending on hardware and Software configuration.

CONTROLS

Controls are used to enter information into the Software. Some controls might become disabled during experimental acquisitions. Disabled controlled are normally shown grayed.

The Software uses the following system of controls, all accessible with a single click of the mouse left button.

-  RUN CONTROLS. Clicking this type of control will activate a process or open a related dialog. This will execute immediately or (in some cases) after completion of a conflicting active process. Clicking an active control again will in most cases cause a process to be stopped (e.g. an experimental run in the main window, or laser excitation in Setup). After clicking, these controls will glow light green until completion of the process or deactivation.
-  ENABLING CONTROLS. Clicking this type of control will permit an operation or function to be performed when a related run control is activated. Such enabling controls will glow green when selected. Controls that select one of two parameter values (such as High or Low Gain for the NIR detector) appear blue instead of green.
- 

NUMERICAL CONTROLS. The number shown in these control fields can be entered directly from the keyboard, or changed digit by digit by clicking the mouse on the increment/decrement arrows at the left edge of the field. Typed values become active when the cursor is moved to another field; incremented or decremented values are active immediately. Acceptable numerical values may be limited by the Software to avoid inappropriate experimental conditions. Some control values, if changed during a run, may be revised in the current run (e.g.: a new interval time or sequence total time in a sequence acquisition). However, other control values will be changed only after the run ends (e.g.: integration time in an experimental acquisition).
- TEXT CONTROLS are windows in which any combination of characters can be typed using the keyboard. It is possible to copy and paste text into and out of these windows.
- FILE PATH CONTROLS are similar to text controls and share their features. However, they have a Browse Button attached to the text box to aid in file path navigation. File paths and names in these controls may be entered using Windows Explorer, which will open after a click on the Browse Button. Alternatively, a file name can be entered by dragging and dropping from any Windows Explorer window. File Path control titles shown in the main window will change automatically from "Save Origin File" to "Rewrite Origin File" if the specified file already exists. Saving into new files, rather than overwriting, is recommended. No file extension is required, and, in some case, a dot, while not necessary, can be acceptable as a part of file name. File names for text files are treated somewhat differently, because when an incomplete file name is entered it will be automatically extended to a full name (see below for more details about saving files). Checking for existing extended file names works in this case, too.
- TAB CONTROLS allow switching between different window tabs by clicking on the Tab name (e.g.: between "Single Acquisition" and "Sequence Acquisition" in the main window).
- DROP-DOWN OR LIST MENUS are used to select a numeric or text value from a list.

-  SWITCH CONTROLS are used to select between left and right groups of parameters or controls.
- SLIDE CONTROLS are equivalent to numerical controls in which values are changed by dragging a slide bar (normally with the mouse).
- COLOR CONTROLS allow selection of colors from a list or through the Windows library.
- Individual elements of arrays and other combinations of controls generally act in the same way as simple elements.

INDICATORS

Indicators do not allow input from the user, but in many cases the user can select which values are shown and/or the display form. Adjustment of display style is especially useful for plot indicators.

- NUMERIC INDICATORS shows numeric values of specific parameters.
- TEXT INDICATORS are windows containing text. This text can be copied, but cannot be modified by the user.
-  BINARY INDICATORS have a square shape and signal two states. Dark indicators are inactive; bright ones are active. Their colors may vary. If an indicator is used to alert a user to a potential problem, the alerting color will be red.
- PLOT INDICATORS are used to graph spectral and kinetic data. When the cursor is inside the plot, the right mouse button accesses a rich menu of plot properties that can be changed by the user. If a plot axis is set for manual scaling, then the display limits for that axis can be specified by double-clicking on the first and last tic labels and entering desired numerical values.
- 3D PLOTS in the present version cannot be changed in the same way as 2D plots. However, powerful mouse controls are available: rotate the plot by mouse motion while holding down the left button; shift the plot by mouse motion while holding down the left button and the Shift key; zoom the plot by rotating the mouse wheel. Many important display choices may be selected though the **3D Properties** dialog in the Software. Note, however, that this dialog cannot be opened during data acquisition. Properties selected for the 3D plot in the main window will also apply to the full-screen 3D display.

7. Primary Mode (Data Acquisition) Operation

The Main Window has three tabbed screens:

SINGLE ACQUISITION TAB

The first tabbed screen, **Single Acquisition**, is shown below. The left side contains graph boxes for displaying emission spectra (top) and absorption spectra (bottom). The top right quadrant (**Acquisition**) shows selection buttons and parameter boxes for controlling the data

acquisition process. Screen objects below this, in the **Results** box, allow selection of file storage and analysis options. The **Instrument Log** box displays real-time information about the steps in the data acquisition process. Finally, the bottom right section of the screen has an action button to leave the primary data acquisition mode and enter the **Setup** mode, another to **Start** the data acquisition process with the current specified parameters, and a button to **Exit** the NanoSpectralyze program.

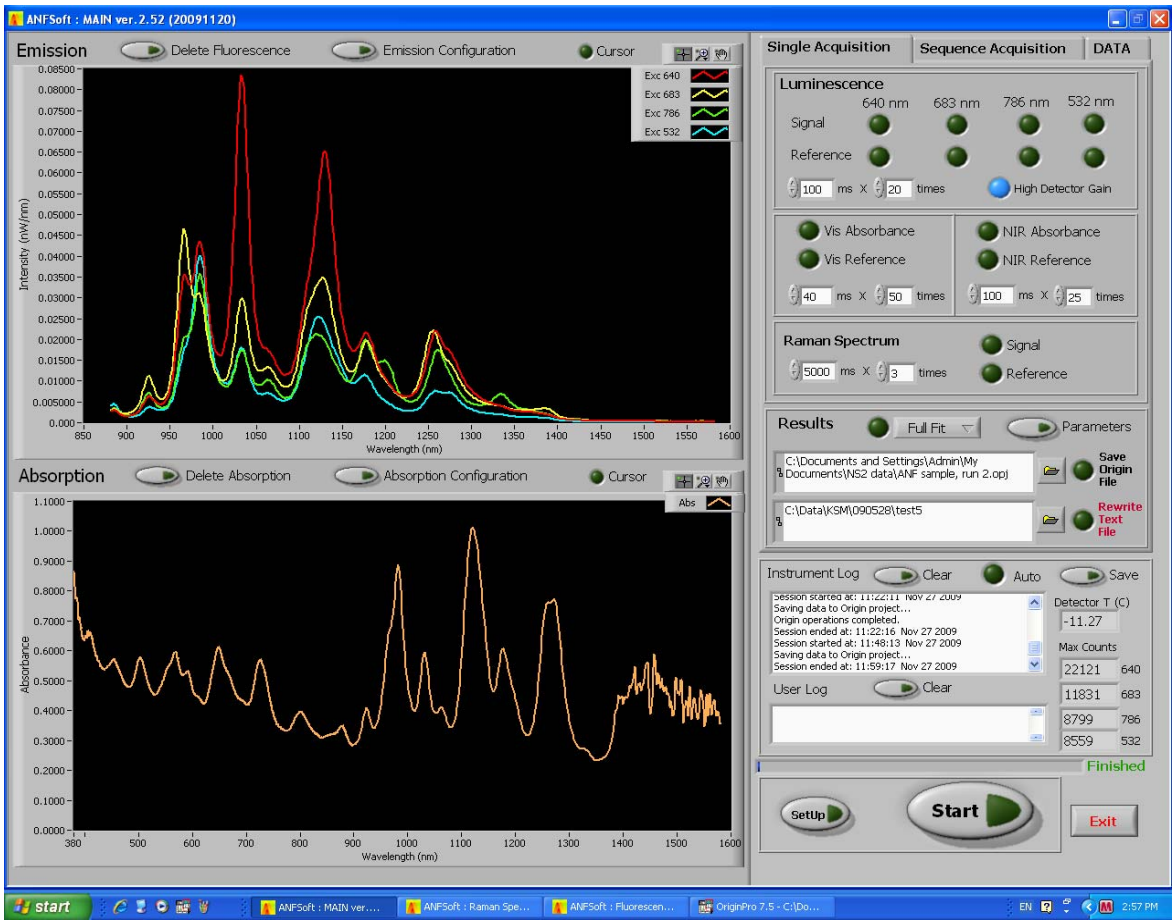


Fig. 6. The Main Window with the Single Acquisition tab selected. The sample contained HiPco SWCNTs dispersed in aqueous sodium cholate.

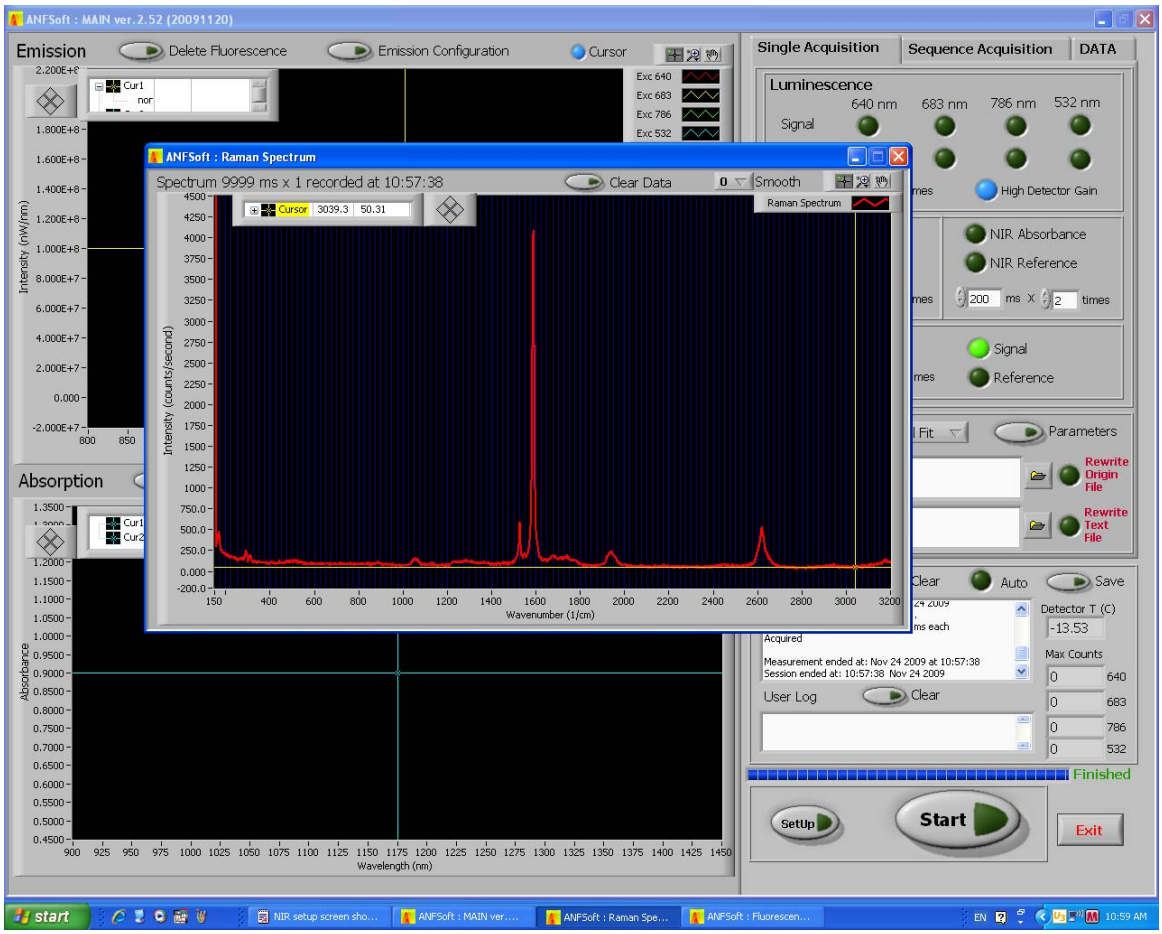


Fig. 7. The Main Window with the "floating" Raman spectral plot window positioned in front of the other spectral plots.

Acquisition Control Group

- Fluorescence spectra may be acquired using any of the installed laser excitation wavelengths. Absorption spectra (as base 10 absorbance values) may also be acquired in the near-IR and (with the corresponding hardware option) visible regions. Select the enabling control buttons next to all of these modes you wish to measure on your sample. The buttons will glow green when selected. In addition, reference data (normally from a blank sample containing no nanotubes) are required for all of these measurements. The reference enabling controls are automatically activated when making the first measurement after starting the Software. Once the reference data have been measured, they remain stored in memory until the Software is closed, and by default they are automatically reused to simplify and speed up measurements on similar samples. However, reference spectra can be re-measured as wished simply by selecting the corresponding **Reference** enabling buttons. For the most accurate measurements, very

freshly acquired reference data are recommended. Note that an information text tip showing when the current reference spectrum was acquired will appear when the mouse cursor hovers over the corresponding reference button. If the reference was imported from a file, this will also be displayed in both single acquisition and sequence panels. The most reliable measurements will require matched integration times for sample and reference.

- Fluorescence may be measured with the detector set for HIGH or LOW gain by toggling the **NIR Fluorescence gain HIGH** button. The button glows blue for HIGH gain. HIGH gain is normal for nearly all SWCNT samples. Very strongly emitting samples may be measured with LOW gain at correspondingly longer integration times. If the gain setting is changed, a new reference measurement is required and will be automatically included in the data acquisition procedure. NIR absorption measurements will automatically be made with the NIR detector switched to LOW gain, regardless of the value set for fluorescence.
- Data **Integration time** (in milliseconds) is entered separately for fluorescence, NIR absorption, and visible absorption measurements. The **Averaging** entry specifies the number of those integration intervals that will be averaged together during a data acquisition run. In practice, the maximum integration times are limited by dark current and the detector range. Because 16-bit analog-to-digital conversion is used in the NIR detector, the maximum signal amplitudes are ~60,000 counts. Following a data acquisition run, the peak raw fluorescence signal counts are shown in the main window for all active excitation lasers. Acquisition times exceeding two seconds are not recommended even for weakly emitting samples. Instead, many shorter acquisitions should be averaged together (using the **Averaging** entry) to attain the desired signal-to-noise ratio. If a possible over-range condition is detected during a fluorescence measurement, the Software will alert the user once. Over-ranging is also possible during absorption measurements. The **Setup** mode can be used to quickly check the raw fluorescence counts and the raw absorption background counts. If a non-standard sample requires adjusted absorption integration times, appropriate values can be chosen in **Setup** mode (see below).
- Selecting the **Absorption Configuration** control button (located between the two data display frames) brings up the dialog box shown below:

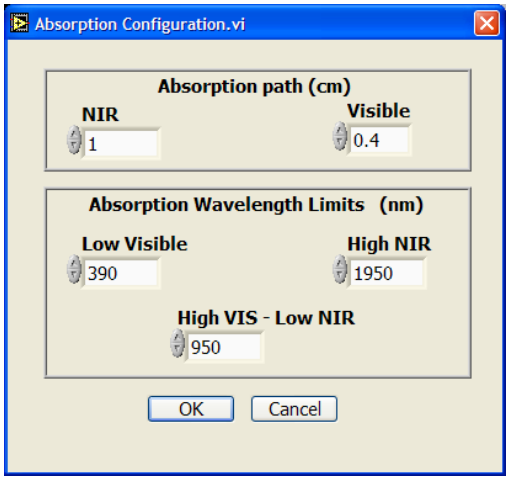


Fig. 8. Absorption parameters dialog window.

This box allows entry of display parameters for absorption spectra. Enter **NIR and Visible Absorption path** values that match the optical path length of your sample cell. Displayed spectra are then normalized to path lengths of 1.0 cm. If no sample dimension is available, the user should use 1 cm path and remember that this resulting absorbance will be per sample. The **Absorption Wavelength Limits** set the plot range of the spectral graph in the lower frame, and the **High VIS – Low NIR** entry specifies the "splice" wavelength for display of the overlapping visible and NIR segments on this screen. Note, however, that the complete overlapping data sets will be saved in Origin project files and text files. If a wavelength entry (such as **High NIR** in Fig. 8) falls outside of the detector's range, the spectrum will be still be limited by the actual detector range.

Results Control Group

The enabling controls in this group will activate processes following data acquisition. These processes may include data saving and analysis.

- The round **Fit** enabling control button on the top line of this box can be selected to order determination of the sample composition through numerical analysis of fluorescence spectra. The button will glow green when analysis is enabled. If fluorescence data are acquired in a run with the **Fit** button enabled, those spectra will be immediately analyzed. If no data are acquired in a run with the **Fit** button enabled, the spectra acquired most recently or imported from a file will be analyzed instead. To import data from files, see the **Import/Export** section below.

- **Full Fit** and **Quick Fit** are the two analysis choices available through the adjacent pull-down menu.

  In **Quick Fit** mode, the spectral fitting process starts and runs automatically using the preselected set of Global Fit Parameters. If the parameters are set for no variation, then (*n*,*m*) species amplitudes will be instantly calculated using the current SWCNT template and an Origin file will be created with the results. If some parameter variations are permitted, the calculation will proceed for one loop and an Origin file will then be created with the results. Quick Fit is appropriate for running samples with spectral properties (reflecting surfactant choice and processing protocol) that are properly represented in the chosen analysis template. Then the (*n*,*m*) distributions can be immediately determined and compared.

  In **Full Fit** mode, the fitting parameters and variation tolerances are first displayed for user inspection and adjustment before the analysis computations begin. Many loops of fitting can be performed if needed to attain an accurate fit. It is also possible to make manual adjustments to parameters between loops. In this mode, data and/or templates can be saved. See **Analysis** below for more details.

- The adjacent **Parameters** button opens an advanced **Global Fit Parameters** dialog window (Fig. 9) to let the user select a template with appropriate spectral parameters and specify the extent to which each of them may vary during analysis. This allows one to tune the data analysis to match a surfactant environment or sample condition. Advanced users can also use this feature to detect and quantify changes in spectral properties that may be induced by physical or chemical changes in a sample.

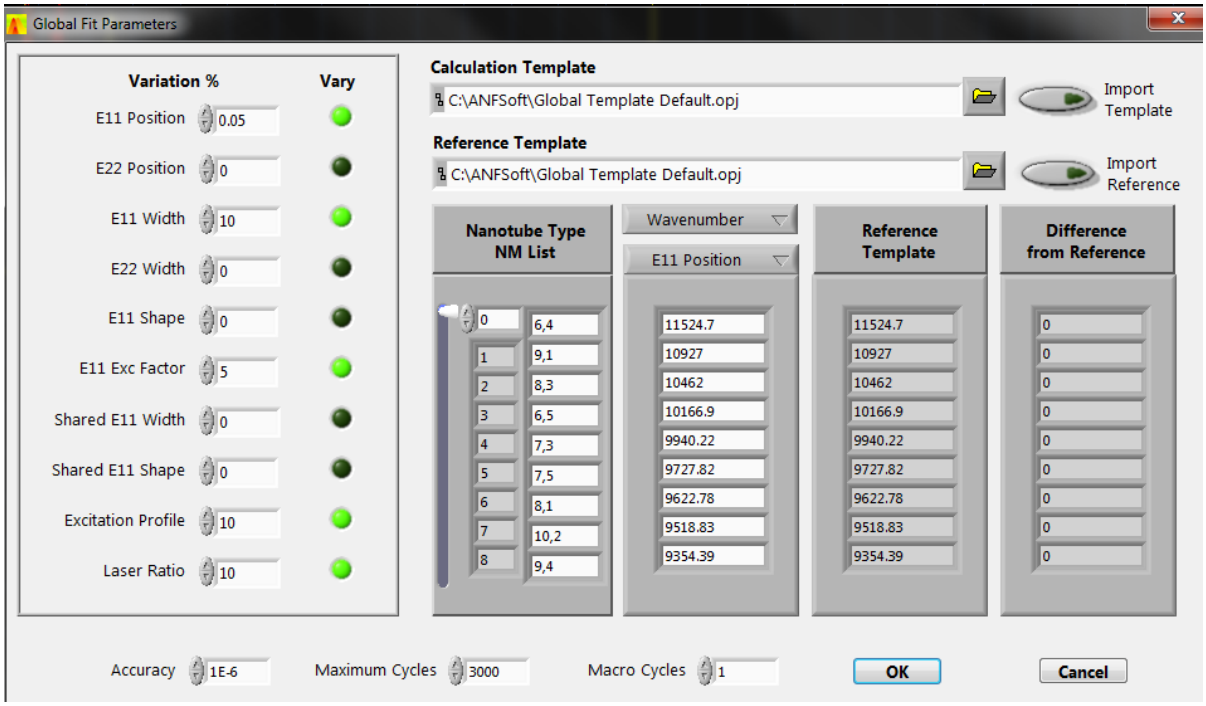


Fig. 9. Global Fit parameters dialog window.

The fields in the large box on the left allow you to enter the allowed ranges of variations (as a percentage of the parameter value) for many fitting parameters. The adjacent enabling control button toggles disabling and enabling variations of the parameter in the calculation. When it glows green, variation is enabled but limited to the relative percentage change in the adjacent field. If a variation range is entered as zero, the green control button will switch off when the calculation begins. For information on definitions of the fitting parameters and guidance on their recommended variation ranges, see **Appendix 5**.

The **Accuracy** and **Maximum Cycles** parameters control the convergence tolerance and duration of the fitting calculation, which uses a modified Simplex algorithm. **Accuracy** represents a convergence criterion equal to the relative difference between the best and the worse dataset points used in the fitting process. Because the default value (0.001) is very small, in most cases convergence will not be reached with this value. In such cases, the current calculation round will stop instead after the selected number of **Maximum Cycles**, or by a manual stop command (see **Analysis** section below).

Fitting requires a **Calculation Template** file with appropriate spectral parameters. During Software startup, **Global Template Default** is loaded. This file contains the last template loaded or calculated before the Software was closed. Alternatively, a different template (or a results file from a prior fit) can be used by entering its name in the file path control and then selecting **Import Template** to load the file parameters. Any Origin project file previously saved by the Software contains embedded template data, including parameters deduced in the fit performed when it was created, and it can be therefore be imported to use as a template for new data fitting. However, the recommended practice is to instead use special computational template Origin files, which are much smaller and can be saved from inside **Global Fit** (below) with clear descriptive names. At present, the default location of template files is the main program folder (ANFSoft), although any other folder can also be used. Templates may be also modified in Origin and then saved. This method can be useful for creating a completely new template or manually making major changes to parameters (such as shifting all peak positions at once).

The lower right quadrant shows tables in four columns. The first is a list of (*n*,*m*) species included in the selected spectral fitting template. The heading on the second one is a pull-down menu that lists each type of fit parameter. When one of these is selected, a list appears below showing that parameter's current value or values. If the selected parameter varies with (*n*,*m*) species, then the adjacent left-hand table shows the corresponding (*n*,*m*) values. The lists can be synchronously scrolled using the slider control on the left. Note that any parameter may be manually revised by an entry into the appropriate field. At the end of a fit, the parameter called **Solution Vector** contains the deduced emission amplitudes of all (*n*,*m*) species in the spectral basis set. Refer to **Appendix 5** for descriptions of other fit parameters.

To monitor deviations of deduced fit parameters from their standard or initial values, enter a file name in the **Reference Template** field and click on the **Import Reference** button to load it. The reference parameters will then be shown for all (*n*,*m*) values in the third column labeled **Reference Template**. The fourth column, **Difference from Reference,** shows the

numerical deviations of the current fit parameters from those in the Reference template. The drop-down menu over the second column allows inspection of differences in any parameter (in nm or cm^{-1} units), and the vertical slider control on the left of the **Nanotube Type** column allows inspection of any (*n*,*m*) value in the fitting basis set.

- The **Save Origin File** enabling control can be selected to record the Origin file containing the spectral data and analysis results (if applicable) into a project file saved to the computer disk. The desired file name should be entered into the adjacent file path control. Using the Browser Button is the recommended way to select a name, because this prevents mistakes with folder names. Also, although new folders cannot be created by typing a new name in the path control, they can be created using the Explorer window. Drag-and-Drop of a file name from an independent Windows Explorer panel is also possible, and the file name can be edited after such selection.
- **Save Text File** enabling control can be selected to record the spectral data as simple text files, one spectrum per file. Similar to **Save Origin File**, the desired base filename should be entered into the adjacent file path control. This base name will be used for all spectral files (e.g. six files in the case of four fluorescence and two absorption spectra), and even for a log file. Note that the base file name will be extended to reflect the specific types of data, as follows. The base name will be appended by 8 symbols "**_???.txt"** (underscore and 3 characters after the name, plus ".txt" for the file extension). For emission files, the three characters will contain the excitation laser wavelength in nanometers; for NIR absorption files, the label "NIR" will appear; for visible absorption files, the label "VIS" will appear; and for log files, the label "LOG" will appear. During saving, only non-empty files will be saved, so if some data were not acquired, the corresponding text files will not be created. Unnecessary spectra can be also removed from the Software using **Delete Spectra** or **Delete Absorption** dialogs activated by controls above the fluorescence and absorption plots.

Status group

The lower group of controls and indicators is the same for single spectral acquisitions and for sequence acquisitions.

- The large text field is the text indicator **Instrument Log**. Real-time messages appear noting the stages and details of the current data acquisition run. During an experiment it scrolls automatically to display the most recent lines.
- The second text block is the **User Log** text control. This is intended to hold the user's notes about the current acquisition run. It is essentially a simple electronic notebook useful for describing the sample or any other information that helps to document the experiment.
- **Clear** is a control that can be used to remove the content of the **Instrument Log** or **User Log**.

- **Save** is a control that forces saving of a log file. The file name is formed by the same system as spectral files, with "_LOG" at the name end. The log file is a text file containing the contents of the **User Log** followed by contents of the **Instrument Log**.
- **Auto** is an enabling control that allows automatic saving of the log file after every experiment. Additionally, the **Experiment Log** will be automatically cleared before the experiment begins. The user log can only be changed manually.
- **Detector Temperature** displays the temperature in degrees Celsius of the InGaAs spectral detector. It should remain quite stable and close to the target set point entered in **Setup**. The recommended set point is normally near -15 °C.
- The **Max Counts** boxes indicate the maximum raw signals in the current emission spectra excited at all laser wavelengths. These values allow quick assessments of fluorescence signal strengths under different sample and integration conditions. If any value exceeds 50,000, the integration time should be reduced (Excessively high values will normally be announced once in a pop-up dialog box during data acquisition).

Bottom Action Control Buttons

- Clicking the main **Start** button begins any enabled combination of data acquisition, data analysis, and data saving. During data acquisition, the button label changes to **Stop** and its function changes to allow a run to be aborted. If **Stop** is pressed during a single spectral acquisition, the unfinished measurements will be discarded. In a sequence run, the last unfinished spectrum will be lost but spectra already measured will be retained.
- The horizontal progress bar above **Start** roughly indicates the completion status of a single experimental cycle.
- **Setup** temporarily exits the Primary (data acquisition) mode and enters the **Setup mode**, described below.
- **Exit** is selected to close the Software session. It is equivalent to closing the main window. A confirmation dialog appears to prevent unintended exits.

Data Display Frames

- The top frame in Fig. 6 shows overlaid, color-coded **Emission** spectra acquired with all active excitation lasers. Only active data are displayed, so if just one spectrum has been acquired, it will be displayed on its own. However, spectra can be acquired in any sequence, and the second or the third one can be added later. After acquisition, the software will only update spectra of the same type (same absorption spectrum or fluorescence with the same excitation wavelength), while retaining the other spectra. Spectral data plotted in this frame have been processed by blank subtraction, correction for the wavelength-dependent sensitivity of the detection system, and correction for detector response nonlinearity. They are displayed by default on an auto-scaled amplitude axis. The plot frame has several standard controls that allow interactive adjustment of auto/manual scaling, axis limits, trace appearance, zooming, etc. To access the menu containing these controls, place the cursor inside the frame and click the right mouse

button. Note that after an acquisition, the number of fluorescence spectra cannot exceed the number of excitation wavelengths, but more fluorescence spectra can be displayed after data **Import**.

- The lower graphic frame shows a plot of the measured sample **Absorption** spectrum, computed relative to the blank cell as a zero-absorbance reference. The plot is also auto-scaled by default, but axis limits can be adjusted by the user after switching to manual scaling (right-click inside the frame, de-select y-axis (or x-axis) autoscaling, and then type the desired axis limits onto the first and last tic labels). Once the physical cell dimensions have been entered into the **Absorption Configuration** dialog box, absorbance values are computed and displayed for a sample path length of 1 cm. This calculated absorbance per cm will be also saved in data files.
- Above each graphic frame is a **Cursor** control button that glows blue when active. This draws up to two vertical and horizontal cursor lines over the plotted spectral data and displays a control box in the upper left hand corner showing the spectral and amplitude coordinates at the cursor intersections. The cursors can be moved by dragging with the mouse or by clicking the four triangular controls located to the left of the **Cursor** button. This feature allows easy examination of peak positions and heights.
- **Delete Spectra** and **Delete Absorption** controls will open the **Delete Emission Spectra** (Fig. 10) and **Delete Absorption** (Fig. 11**) boxes**. Manual deletion of spectra can be useful to avoid displaying mixed data when switching samples, or when importing several spectra. Any number of imported fluorescence spectra may be displayed, but the current Software version will display only one set of VIS-NIR absorption spectra. To delete fluorescence spectra click the enable control next to each file to be deleted and then **Delete** (Fig. 10). If the list exceeds the window size, a scroll button will appear. Clicking on **Delete All** will remove all fluorescence spectra from the display. The **Delete Absorption** dialog (Fig. 11) allows removal of one or both spectra.

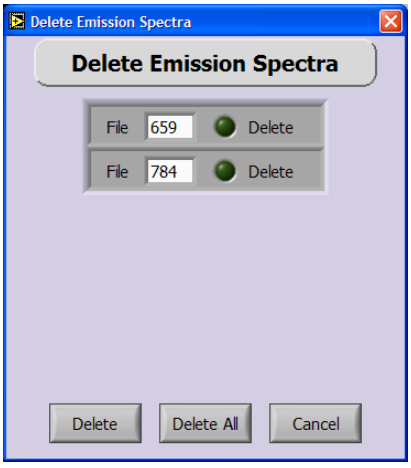


Fig. 10. Delete Emission Spectra Dialog

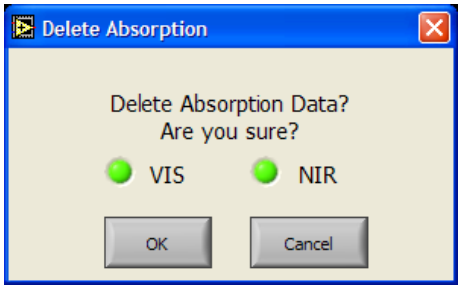


Fig. 11. Delete Absorption Spectra Dialog

- The **Emission Configuration** button above the Emission frame activates a dialog box that permits display of emission spectra in one of three spectral units: nanometers (nm), wavenumbers (cm^{-1}), or electron volts (eV). The emission amplitudes may be displayed in units of power (W per spectral interval) or photon flux (photons per second per spectral interval). The default display style, Spectral power vs. wavelength, will always res
- The **Fluorescence Power and Efficiency** results box (shown below) will appear following single data acquisition. The spectrally integrated emission signal is displayed on the **Total Power** line for each of the excitation wavelengths that was used. These values are corrected for the detection system's wavelength dependent response and are given in units of nW of optical power at the aperture of the optical fiber that collects the emitted light. If the sample's visible absorption spectrum has also been acquired, the Total Power values are divided by sample absorbance at the corresponding excitation wavelengths to give the **Efficiency** values listed on the line below. The units here are nW per inverse cm, or nW cm. These values can be viewed as proportional to the sample's quantum yield for near-IR emission. They provide a valuable measure of the fraction of the sample content consisting of emissive SWCNTs. Note that impurities and nanotubes that are defective, bundled, or significantly derivatized will contribute to absorption but not to emission and will therefore lower the **Efficiency** values. Higher **Efficiency** values will be found for samples containing large fractions of disaggregated pristine SWCNTs. The numerical values shown in the **Fluorescence Power and Efficiency** results box are automatically recorded in the log file for the experimental run.

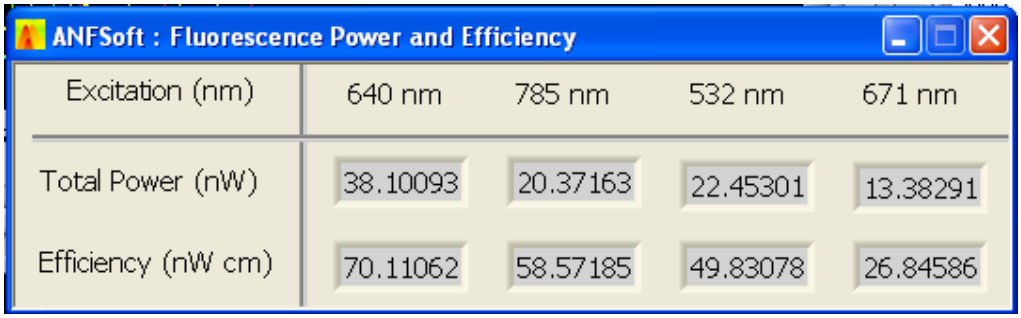


Fig. 12. Fluorescence Power and Efficiency results box.

SEQUENCE ACQUISITION TAB

The second tabbed screen of the Primary Mode is **Sequence Acquisition**. This sub-mode allows automatic measurement of spectral data at specified time intervals and flexible display of the resulting data sets. Typical applications include monitoring the stability of SWCNT suspensions over time, studying the kinetics of chemical and physical SWCNT reactions through spectral changes, and monitoring eluent streams containing SWCNTs (using a flow cell in place of the static sample cell). The NanoSpectralyzer has the ability to make nearly concurrent kinetic measurements of absorption (visible and/or NIR) and fluorescence during such sample transformations.

The **Sequence Acquisition** screen is shown below.

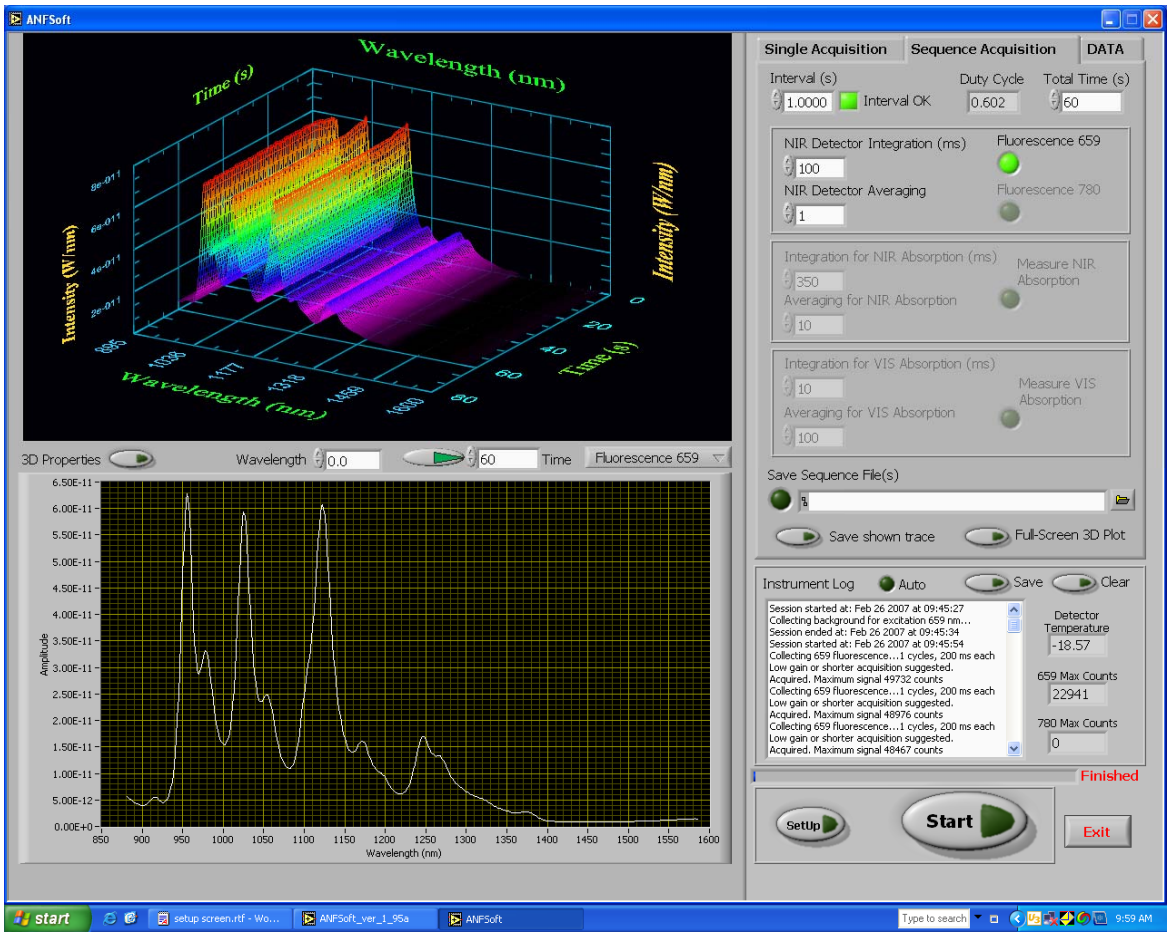


Fig. 13. Sequence acquisition screen.

Acquisition group

- To enable a sequence acquisition, first acquire the corresponding reference spectra using the **Single Acquisition** screen, as described previously. Then choose the desired combination of fluorescence and absorption spectra by selecting the corresponding enabling control buttons, which glow green when active. The controls will be gray and disabled until the corresponding reference spectra have been acquired in **Single Acquisition**.
- On the **Sequence Acquisition** screen, enter into the **Interval** numeric control box the intended period in seconds between repeated measurements. This is the target delay time for acquired spectra (see **Duty Cycle** below).

- In the **Total Time** box, enter the total duration of the run, in seconds. Acquisition will stop after this time has elapsed. Note that this value can be increased or decreased during the run, and acquisition can also be stopped manually at any point.
- The parameters entered into the **Integration time** and **Averaging** control boxes are equivalent in function to those in the **Single Acquisition** screen. However, parameter values for single acquisition and sequence acquisition mode are completely independent. The use of an integration time value equal to that used for the corresponding reference spectrum is highly recommended. (The current reference integration time is displayed in the "tip strip" text activated by placing the cursor over a control box.)
- The **Duty cycle** box displays the fraction of each interval needed for measurements. If this value is less than 1, the square **Interval OK** indicator glows green. This means that the system should be able to achieve the requested **Interval** using the specified acquisition parameters. Otherwise, the indicator will be red, warning the user that the requested **Interval** is unlikely to be achieved. If a stable **Interval** is important, this can be corrected by reducing the **Averaging** or **Integration time** parameters, and/or decreasing the number of spectral types to be acquired. Note that even if the Duty cycle indicator is red, it is still possible to make measurements. In this case the Software will acquire all requested spectra with requested integration times and averaging at the maximal possible rate; the actual times at which spectra were measured will be properly recorded in the data file, even if the intervals are somewhat irregular and exceed the requested interval value.
- To perform the fastest sequence measurements, (1) select only a single type of spectral acquisition (switching between different excitations and absorption creates a delay), and (2) disable the 3-D live plotting for large runs. To disable live plotting, select an unused spectrum type via the rectangular menu button found under the bottom right corner of the 3-D plot frame (see below **Display Group**). Even though the pull-down menu will show unused types as grayed out, they can be selected in order to blank the display and speed up data acquisition. This acceleration is especially significant for single-processor computers. Under these conditions an acquisition rate of more than 10 spectra per second should be possible with a short integration time and an averaging parameter of 1.
- As in single acquisition experiments, the **Start** button at the bottom of the screen is used to begin data acquisition and to save data after the run (if enabled).

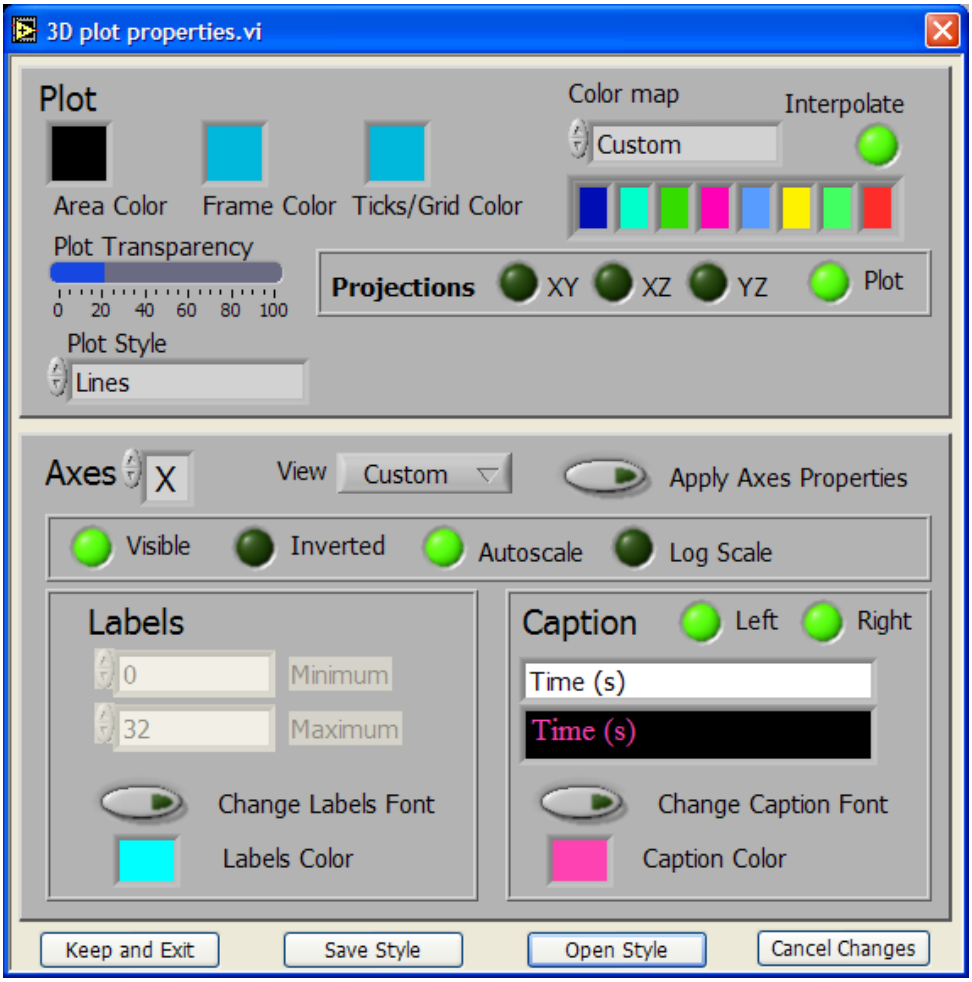


Fig. 14. 3D Plot Properties dialog. Type of 3D plot, colors, axes captions and labels and many other parameters can be modified, saved and restored.

Display group

- The upper left quadrant shows a 3D plot of spectral data as a function of time, updated during the acquisition process. By clicking and dragging with the mouse, the orientation of this plot can be varied at any time, including during data acquisition. If the ALT key is held down, the mouse will zoom the plot. If the SHIFT key is held down, the mouse will shift the plot within the window.
- After system start, the design of the 3D plot is loaded from file **3D plot Style.3DT** which was saved after the last view change. Very detailed control over the appearance of the 3D plot is available through the **3D Plot Properties** dialog box accessed by clicking the **3D Properties** button. It also allows saving and loading other 3D styles, for easy changing from color to black and white and other surface types. Several styles are usually provided with the software.

- To select which spectral data are displayed in the 3D window and the spectral or kinetic slice (see below), select the type of spectrum from the pull-down menu on the right between two graphic panels. Unavailable spectra will be listed in gray, but can still be selected to empty the graphic plots. **Note: even if only a single spectral measurement is underway, the display selection does *not* automatically default to show that measurement** (this allows the fastest acquisition). **You must select a spectrum from the pull-down menu in order to observe the 3D data plot and 2D slices.** The display selection can be switched during a run.
- The lower left plot contains a kinetic or spectral "slice" of the 3D data shown above. To view a kinetic trace at one wavelength, click the central toggle control so that its green arrowhead points to the left, to a wavelength mark. Then enter the desired spectral value into the **Wavelength** numeric control box. To view a spectrum at specific time, click the central toggle control so that its green arrowhead points to the right, to the time mark. Then enter the desired time value into the **Time** numeric control box. Values of **Time** and **Wavelength** can also be changed in advance. The 2D slice display can be changed during a run.
- When the **Time** and **Wavelength** parameters are changed manually (by typing or using the numeric control increment-decrement button), a requested value might not match any available in the data set. In such cases, data corresponding to the closest available value of that parameter will be displayed.
- By default, **Time** values for the 2D spectral display will increment automatically during an experimental run as new data are acquired. This will continue so long as the **Time** box contains the maximum (current) value. Optionally, other slices can be displayed during the run by manually entering the desired time or wavelength value. The user can return to the automatically updated 2D spectral display mode simply by entering any large value into the **Time** box (forcing it to the current maximum).
- During sequence acquisition, the current number of acquired spectra is displayed and updated on the left of the screen between upper and lower plot windows.
- A data slice displayed in the lower left frame can be saved by clicking the **Save shown trace** button under the **Save Sequence File(s)** button. A dialog box requesting the new file name will then appear.

Saving group

- After a sequence run is complete, the data display can be expanded to full-screen by clicking **Full-Screen 3D Plot**. Controls at the bottom of the full-screen display allow adjustment of axis scales, and mouse controls for zooming and rotation remain active. The plot style will be the same as in the smaller 3D plot. Full-screen plots can be easily copied to the Windows clipboard (using the keyboard command Alt-PrintScreen) for insertion as a picture into any document.
- To enable sequence data to be saved after the run, enter a file name into the **Save Sequence File(s)** box and select the adjacent button, which glows green when activated.

As for single spectrum files, file names will be automatically extended to code for the type of data that they contain. Saving can be requested automatically after the run, or later, after the data have been acquired and examined. The output file is a plain text (ASCII) file with the structure XY-many Z.

- The **Save shown trace** button will save the displayed 2D spectral or a kinetics plot. A full file name (not a reduced form) should be entered by the user in the dialog box that opens.

DATA IMPORT AND DISPLAY TAB

The third tabbed screen of the Primary Mode is **DATA.** This screen allows flexible import of previously acquired spectral data that have been saved as text files, or as spectral sequences, or as Origin project files. When importing files generated by programs other than the Software, users should insure compatibility of file structures and should use consistent naming conventions to distinguish file contents.

- The **Import Spectrum** dialog (Fig. 15) should be used to load ASCII text files containing spectra into the Software. One set of absorption spectra can be loaded, marked as NIR and VIS. These labels are shown in the **File** text controls on the left. In order to be imported as a fluorescence spectrum, the file label entered should be a 3-digit integer. Import is not limited to data files acquired by the Software. However, import is optimized for such Software-generated files, and all fluorescence and NIR absorption files should have the same X-scale, which is interpreted as wavelength (the first column of the text file). The recommended import sequence includes entering a file name into the first line (via Browse Button or by dragging and dropping a file from any Windows Explorer window – especially convenient with two monitors). The file need not correspond to the file label on the left. If this is a file named with the standard convention, a user can click the **Find Other Files** button, and all files with the corresponding name will populate the dialog box. The wavelength portion of the name will automatically appear as the file label, and import of all files will be enabled. To selectively disable import, just deactivate the adjacent control button. The drop-down menu on the top left provides control of import options. **Replace all** will delete all current spectra in the Software and replace them with a new imported set (if it is not empty). **Replace** will replace the last file with the same excitation wavelength label if a new file with this label is imported. **Append** will add a new file to the list (and to the spectral plot) following other files with the same label. The **Append** option is useful for quickly overlaying and comparing fluorescence spectra acquired in different experiments.
- The **DATA** screen is shown below with an open dialog box for importing text (ASCII) data files. Note that the **Import Spectrum** and **Import Sequence** functions can read files from any source (not just those acquired by NanoSpectralyzer) as long as the data are written in compatible two-column ASCII form. NanoSpectralyze can then be used to display and analyze the data.

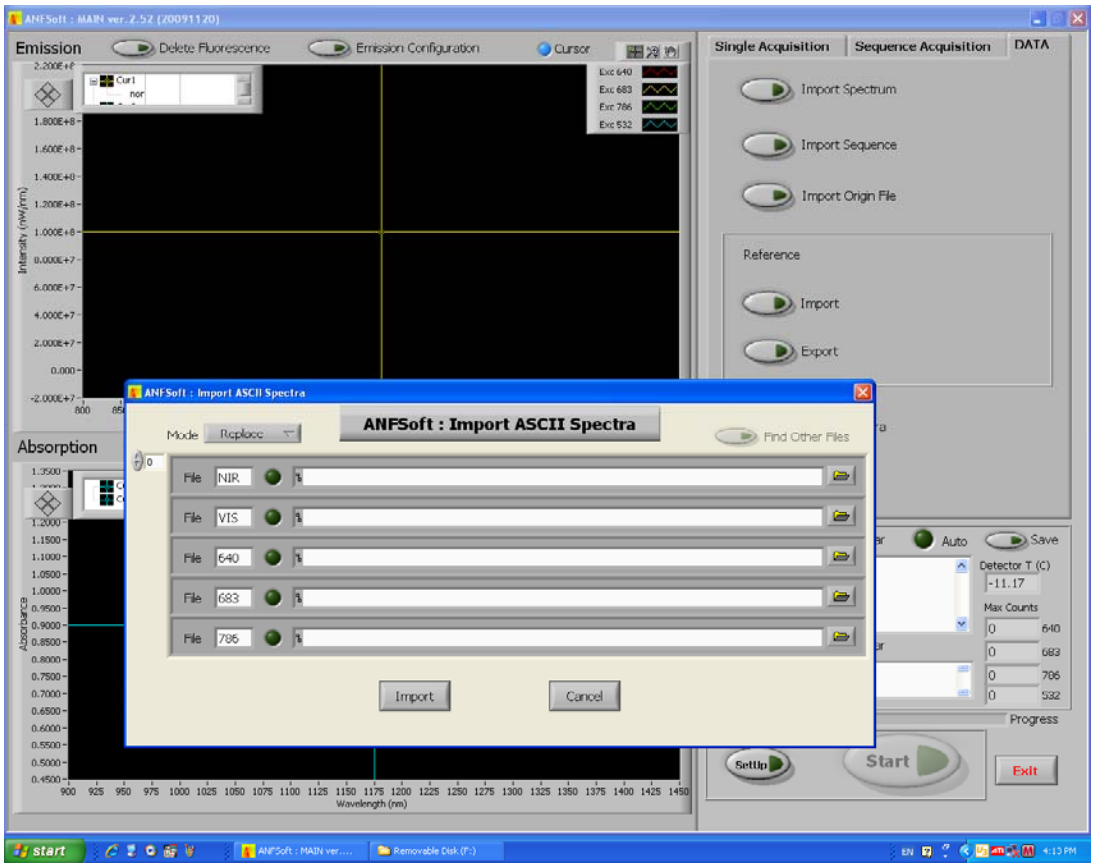


Fig. 15. DATA screen with an open dialog box for importing text (ASCII) data files.

- **Import Sequence** is very similar **to Import Spectrum**, and it uses the same dialog. Instead of the simple two-column XY text files, files in format XY-manyZ sets are imported. Since there is no good comparison method for 3D plots, the recommended option is Replace.
- **Import Origin File** always replaces the current spectral set with data from an Origin file previously saved by the Software. The file could have been saved with or without fitting, as only the experimental portion will be imported (the calculation template can be loaded in **Global Fit Parameters**). This import is equivalent to the **Replace All** mode of **Import Spectrum**. If some spectra are absent from the Origin file, they will be empty after import.
- **Import / Export Reference** can be used to temporarily save and restore a reference file. This simplifies performing measurements with more than one fluorescence background or absorption reference, as the reference measurement need not be repeated. Imported reference files are identified in the "tip strip" activated by moving the mouse over the reference enable button or sequence enable button.

- **Remove Spectra** opens the same dialog box as **Delete Spectra** in the **Single Acquisition** panel**.**

8. Analysis and Results

Fitting of fluorescence data is enabled from the **Single Acquisition** screen. Selection of **Quick Fit** causes data analysis to be launched automatically with no user action or input. The **Global Fit** window will appear, and fitting will be performed according to parameters selected in the Global Fit Parameters dialog box, which is accessible from the Single Acquisition panel and from the Global Fit window. Selection of **Full Fit** mode causes the system to pause at the start of the fitting process while displaying the screen shown below. The user can then manage the fitting process as described below:

- Clicking the **Calculate** button will open the **Global Fit Parameters** dialog box so that the user can select a template file for the fit, examine the spectroscopic parameters, select parameters to be varied, and set tolerances for those variations. To start the calculation, click **OK**. At the end of a fitting round (terminated by reaching the specified Accuracy or Number of Cycles), clicking the **Calculate** button again will cause fitting to continue for another round, with all parameters starting at their last adjusted values. A round of calculation round can be interrupted before completion by clicking the same control again.

- When the **Redraw New Results** enabling control is selected and glows green (default selection), the graph will be updated during the fitting process as improved intermediate fits are found. Otherwise, a new plot will be displayed only after completion of the fitting round. This control can be switched during a fitting run.

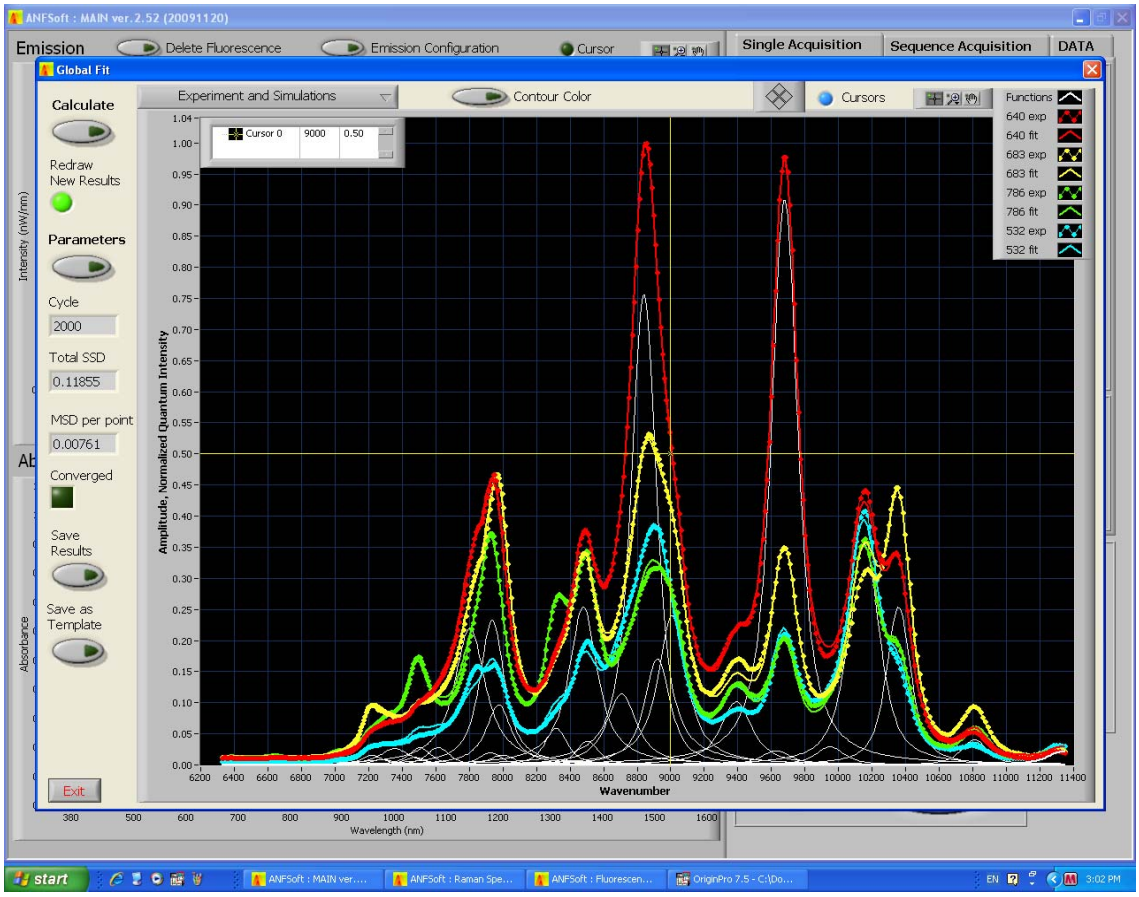


Fig. 16. Fitting of fluorescence data with Quick Fit from Single Acquisition screen.

- Clicking **Parameters** will open the same **Global Fit Parameters** dialog box. However, when parameters are changed, clicking **OK** will accept the modifications without causing a new fitting round to start.
- The **Counts** indicator window shows the number of fitting iterations completed since the start of the calculation. When the number reaches the **Maximum Cycles** selected in the **Global Fit Parameter**s, the calculation will stop. It may stop earlier if convergence to the current tolerance level is achieved. The computational time per cycle is nearly constant, so the **Counts** value will change at a nearly constant rate, except at the very beginning and at some special points in the calculation algorithm.
- The **SSD** indicator window shows the lowest value of the summed squared standard deviations currently achieved in the fitting process. It updates promptly when a better simulation is achieved. If **Redraw New Results** is enabled, all spectral simulation traces will be redrawn, showing their approach to the experimental traces.
- The **MSD** indicator window shows the relative standard deviation between the experimental and simulated spectra, averaged over all data points. This value updates

only at the end of a fitting run. The value is based on the SSD and the number of spectral points (which is constant in any fitting process).

- The **Converged** indicator glows green when the specified accuracy limit is achieved.
- At the end of the fitting process, the final computed simulated spectra will be overlaid with the experimental spectra. The deduced set of (*n,m*) spectral components for the simulated spectrum excited by the first laser (shortest excitation wavelength) will also be drawn at this point.
- If desired, the computation can be continued with the set of parameters that were deduced in the previous attempt and displayed in the **Global Fit Parameters** dialog. These can be changed manually before the next round in **Global Fit Parameters**, or by editing the parameters of a specific (*n,m*) species in the **Simulated Contour** graph display (see below).
- The **Save Results** button opens a dialog box for saving the results (final fit parameters plus data and simulations) to an Origin file.
- The **Save as Template** button opens a dialog box for writing a template file containing only the final fit parameters. This compact file contains no data, but can be valuable for use in future fitting sessions.
- Note that the time needed for spectral fitting will vary strongly with the number of parameters that are allowed to vary. When first analyzing samples prepared with a new protocol, it may be necessary to perform a lengthy and careful fit in which many parameters are varied. After an optimal fit has been obtained, the deduced parameters should be saved to a new template file that can be loaded later to analyze other similarly prepared samples. In those subsequent fits, most parameters can normally remain fixed for much faster computation. If no spectral parameters are varied, only the amplitudes of (*n,m*) species are adjusted to fit the data, and fitting is virtually immediate.
- After the first round of fitting, a drop-down menu selector will appear above the graph window. In addition to the default **Experiments and Simulations** view, it will display of a **Simulated Contour** plot computed from the deduced fit parameters and species concentrations (see Fig. 17 below).
- Keep in mind that this contour plot is a reconstruction deduced from the few fluorescence spectra and the current fitting parameters, so its appearance depends on those parameters and the quality of fit. This display can be very useful for identifying anomalous or unrealistic fit parameters, such as spectral widths that are too small or large, and for correcting such anomalous values. For example, incorrect (usually excessive) spectral widths might be deduced for some (*n,m*) species after many rounds of calculation. Such unrealistic parameters can easily be overlooked when examining 2D fits to emission spectra, such as in the **Experiments and Simulations** display, but they will be readily apparent in the **Simulated Contour** display.

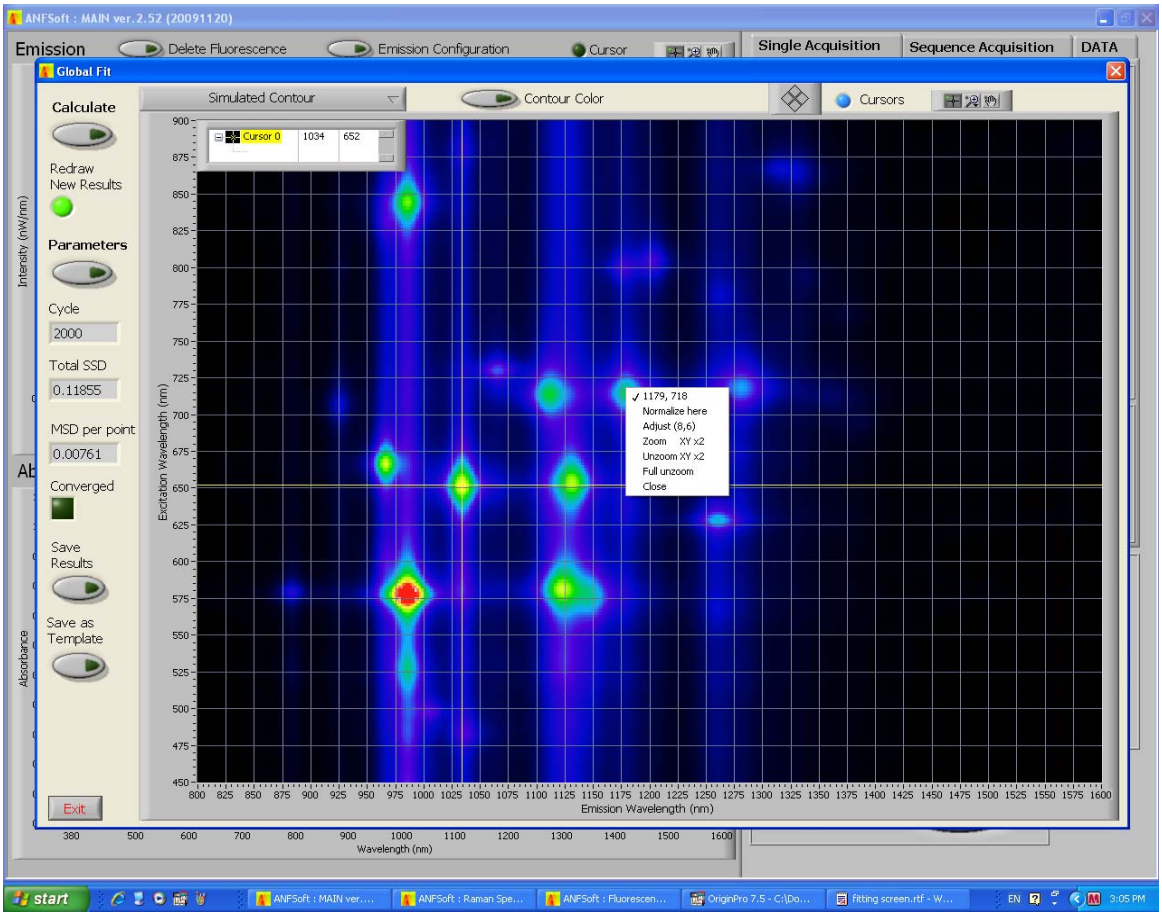


Fig. 17. Simulated Contour Plot of emission intensity vs. excitation wavelength and emission wavelength.

- A right mouse-click in the graph field of the contour plot will show excitation and emission wavelengths at the cursor position (this point will be the upper left corner of the resulting drop-down menu box, as shown in Fig. 17). This menu box contains options for contour plot rescaling (accessible with the left mouse button) and also allows opening a dialog box to modify parameters for the nearest (*n*,*m*) type, which is identified in the drop-down menu. When parameters are modified and the **SWNT Edit** dialog box is closed, the contour plot will be redrawn according to new parameters. The spectral simulation calculation can then be continued with the new parameters.
- The **Contour Color Map** dialog box allows changes to the set of colors used to represent emission intensities in the contour plot.
- Intermediate results can be saved by clicking on **Save Results** after a left mouse-click. A dialog box allowing entry of a file name will then appear. This feature can be used

between calculation rounds to provide a backup record of the current simulation in case manual adjustment of parameters leads to poorer fits in further calculation rounds.

- Clicking **Save as Template** allows a template file (a small Origin project file containing no spectral information) to be saved. This is the recommended method for saving new calculation templates that have been optimized for specific SWNT sample conditions.

It is possible to re-analyze previously acquired data. The active data (data that have been loaded into the program) will be analyzed when **Fit** (but no spectral acquisition**)** is enabled and **Run** is clicked. This process can be repeated to re-analyze the same spectra, even when Global Fit is closed. Spectra saved previously in ASCII or Origin files can be loaded using Import and analyzed in the same way. The last template loaded into the Software will be used as the active template in the calculation.

Structure of the output Origin file

Origin files can be saved with or without spectral fitting. Note that the calculation (fitting) template is also an Origin project file. They have similar structures, but differ greatly in the number of Origin worksheet and graph windows that are contained. The template file is quite small (one worksheet) and contains no experimental data. An experimental file saved without global fitting will be intermediate in size, as it contains experimental data and the template worksheet, but no calculation results. The largest files are created after global fitting, because they also contain the fitting results as worksheets and graphs.

Three types of windows can be found in the projects created by the Software. These are Worksheets, Graphs and Matrices. For more detailed information on Origin usage, please refer to the Origin manual or use Origin Help from within Origin.

The list below describes windows that can be found in an Origin project file. [Note: a numeric three-digit label "nnn" at the end of an Origin window name denotes the excitation wavelength in nanometers corresponding to the data or fit in that window.]

- **DATA1**

  Data1 is a worksheet present in all Origin files created by the Software. Its main content is the template data (information about spectral properties of specific SWNT (*n*,*m*) species), but some extra information is also present (version of the software and some results found in fitting).

- **Data**nnn

  A worksheet containing numerical data for fluorescence spectra acquired with excitation at "nnn" nanometers.

- **Fit**nnn

  A worksheet containing the results of fluorescence spectral fitting for the excitation wavelength "nnn".

- **Index**nnn

  A worksheet used for labeling peaks on a fluorescence plot (only after fitting calculation).

- **Fluor**nnn

  A graph window containing a plot of one experimental fluorescence spectrum, acquired with the "nnn" nanometer excitation laser. In a file created after fitting, it also shows the computed spectral simulation obtained from nonlinear least-squares fitting with individually varied (*n*,*m*) species amplitudes. Spectra of these individual (*n*,*m*) components are also plotted. Note that the spectral axis in this graph is optical frequency (cm^{-1}), rather than wavelength (nm), as is normally shown on the main screen during data acquisition. Peaks arising from the major (*n*,*m*) components are marked with labels near the top of the frame. The plot is based on worksheets **Data**nnn and (after fitting) **Fit**nnn and **Index**nnn.

- **NIR** and **VIS**

  Worksheets containing experimental data for NIR and VIS absorption spectra.

- **Absorbance**
  A graph window showing absorbance vs. wavelength, using data in the NIR and VIS worksheets. This plot will be empty if no absorption spectra were acquired. Overlapping segments from the visible and NIR detectors are shown if the data run included both ranges. The plot shows measured absorbance divided by path length in cm, so as to match the readings when segments involve different cell path lengths. Absorbance values larger than ~3.0 (before division by path length) may be unreliable.

- **RESULTSGF**

  A worksheet created after calculation to hold numerical results of the fitting process. See Appendix 6 for details.

- **HISTDATA**

  A worksheet containing diameter distribution data, as plotted in the **Histogram** graph window.

- **Histogram**
  A column graph window showing a histogram of the deduced diameter distribution of semiconducting nanotubes in the sample. It should be interpreted as a distribution in mass (not number of nanotubes), under the (default) assumptions that the E_{22} absorptivity per carbon atom and the fluorescence quantum yields are equal for different (*n*,*m*) species. This graph is automatically labeled with numerical values of the mean diameter and dispersion about the mean.

- **GrapheneData**

  A worksheet with data used in **Graphene_sheet** plot.

- **Graphene_sheet**

  A graphene sheet plot of the relative abundances of various semiconducting (*n*,*m*) species in the sample. The thickness of the hexagonal border surrounding an (*n*,*m*) label is proportional to the deduced relative abundance, which is again found (by default) with the assumptions of equal fluorescence quantum yields and absorptivities per atom among the species.

- **Sim3D** and **XY**

  Worksheets containing simulated data (Sim3D) and excitation and emission wavelengths (XY) used to create a simulated excitation-emission contour plot.

- **Mat3D**

  A matrix window that contains data used to generate the **ProfileSWNT** graph plot.

- **ProfileSWNT**

  A color-coded contour plot showing a ***simulated*** excitation-emission data scan. It is constructed using parameters deduced from the spectral fitting process and based on the matrix **Mat3D**. Profiles of intensity vs. excitation or emission wavelength are plotted in adjacent frames, and the two cursors designating profile wavelengths may be moved by dragging with the mouse or by typing wavelengths into the section labels. Note that improper fitting or anomalous parameters may cause this contour plot and its profiles to show unrealistic features.

9. Setup Mode

This mode allows simple, real-time checks of sample emission and absorption, with direct control of experimental parameters but without subsequent analysis. Opening the Setup panel will not change any data in the main acquisition panel, as all parameters and acquired spectra will be retained, unless the Setup panel was used to change the system configuration via its **System Parameters** or **Read Parameters** tabs. If a large change in detector temperature set-point is made in Setup, a new NIR background should be acquired. When the Setup screen opens, it accepts detector parameters from the Single Acquisition screen, but those parameters will not be altered by Setup entries. The panel will also retain its spectra until they are reacquired or the Software is exited. In this way, the Setup panel can sometimes serve as an auxiliary acquisition mode that does not disrupt the main acquisition settings. This can be useful for temporary tasks such as checking sample preparation.

Several different display and control screens can be viewed by clicking the corresponding tab selector at the top of the Setup window:

NIR DETECTOR TAB

The **NIR Detector** screen is shown in the figure below. The left side of this screen contains two plot windows. The upper window displays current data as raw detector counts vs. wavelength. Unlike spectra shown in the Main (data acquisition) Mode, these plots are uncorrected for spectral variations in system response. Any spectrum in the upper window can be "frozen" and copied into the lower window for future use by clicking the **Take Copy** button. Clicking this button again will overwrite the data displayed in the lower window.

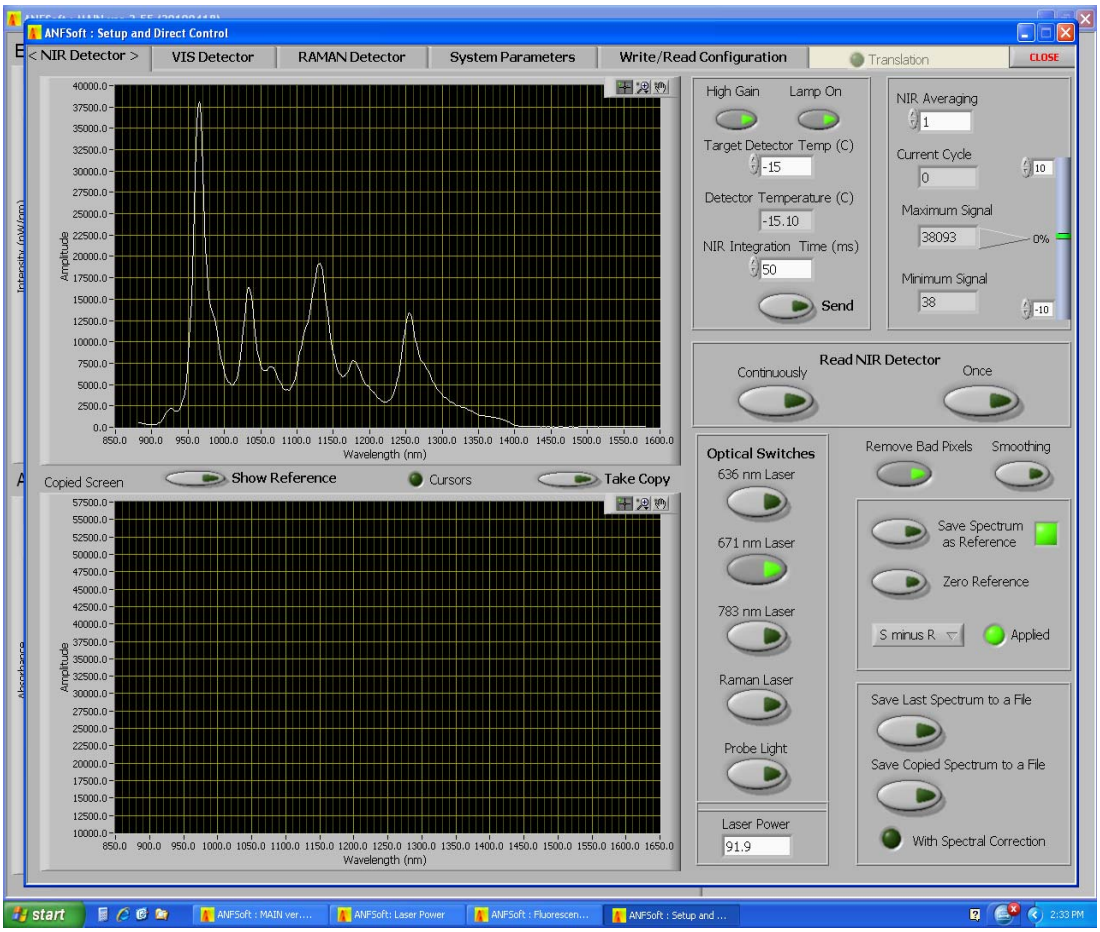


Fig. 18. The NIR Detector screen of Setup mode. The green indicators show that the second excitation laser is active, no acquisition is running, bad detector pixels are removed from spectra, and spectral background subtraction will be applied during acquisition. Laser power in milliwatts is displayed in the lowest box.

The following experimental controls and read-outs are provided:

- **High Gain** button toggles between the high and low gain modes of the near-IR detector. It glows green in High mode.

- The **Lamp On** button toggles power to the lamp used for sample absorption measurements. The probe source is integrated in the NIR detector. For more stable operation, the source is normally kept On during standard experimental sessions. The lamp will therefore automatically turn on when the Setup panel is closed, even if it had been switched Off.
- **Target Detector Temp** may be entered in the box. Lower temperatures provide reduced dark currents, but stable operation may not be possible below approximately -15 degrees Celsius. The target temperature entered here is retained for all modes and shown when the NanoSpectralyze program launches.
- **Detector Temperature** displays the current temperature of the InGaAs detector. Small sensor offsets may make it deviate slightly from the target temperature. A small constant deviation (<0.5 C) does not indicate a problem.
- **NIR Int Time** allows entry of the data integration time, in milliseconds.
- **NIR Averaging** allows entry of the number of integration periods that will be averaged between updates of the displayed spectrum.
- **Send Parameters** must be selected to activate the entered settings. This button will glow green when selected and then go dark after the parameter transfer is complete. ***Note: this transfer cannot occur while the NIR detector is in continuous read mode.***
- **Maximum Signal** and **Minimum Signal** boxes display the largest and smallest intensity values in the current spectrum. The numbers are for the displayed spectrum after all processing (averaging, background subtraction, bad pixel removal, and so on as selected by the user). Note that the intensity is a raw value, not scaled to acquisition and uncorrected by the instrumental spectral sensitivity function.
- The adjacent vertical bar is a graphical display of changes in maximum signal from one acquisition to the next. It shows green for an increase and red for a decrease. The upper and lower limits are adjustable.
- **Read NIR Detector** contains buttons for reading **Continuously** and **Once.** These buttons glow green when acquisition is pending or in progress.
- The **Optical Switches** group contains controls for activating lasers and the probe beam used for absorption measurements. Each beam is independently controlled by its button, which glows green when that beam is activated. Any combination can be selected.
- The text control **Laser Power** displays the measured power, in milliwatts, of the currently activated laser (in its fluorescence excitation role). The text will read "N/A" if no laser is activated or if more than one laser is activated.
- The **Remove Bad Pixels** button can be selected to omit the mapped defective pixels from the spectral display. It glows green when active.

- The **Smoothing** button performs adjacent-channel averaging when selected (this helps to suppress alternate-channel noise from detector multiplexer mismatch). It glows green when active.
- **Current spectrum as Background** can be selected to subtract the currently displayed spectrum from subsequent spectral displays. It is most often used to automatically subtract blank backgrounds. The adjacent round indicator labeled **Applied** glows green when background subtraction is active.
- **Reset Background** can be selected to erase the current background spectrum. The display then shows raw data, including dark current, electronic offsets, plus any light signals that may be present. Note that this button glows green until the reset function is completed.
- The **Save Last Spectrum to a File** button can be selected to write the spectrum displayed in the upper window to a disk file for later use. If desired, select the adjacent enabling control **With Spectral Correction** to save spectrally corrected rather than raw (displayed) data. A dialog box will appear to allow entry of the desired filename. This button glows green until the write is completed. Note that the file cannot be saved until the detector stops reading. If correction is selected, the spectrum will be saved in the same format and spectral power units as spectra from the standard measurement session, with normalization for acquisition time.
- **The Save Copied Spectrum to a File** button is equivalent to the one above it, except that the spectrum in the lower, **Copied Screen**, window is written to disk, also with or without spectral correction as selected.

VIS DETECTOR TAB

The **VIS Detector** screen is shown below. It differs from the NIR Detector screen only by lacking some controls, such as for the lamp and gain settings, detector temperature, and averaging. Note that the maximum integration time for absorption measurements made with the visible detector is often only ~50 ms. Multiple repetitions should be averaged to improve signal-to-noise ratio, but the total acquisition will take longer than estimated because of the overhead time needed for analog-to-digital conversion of all spectral points in the detector before transmission to the computer.

It is also possible to use this screen to check the peak wavelengths of the excitation lasers. Close the flags for VIS and NIR absorption, and place a weakly scattering (milky) liquid into the sample cell. Then when either laser flag is activated, a portion of that beam will be scattered into the visible detection path and its spectral profile will be displayed in the plot window.

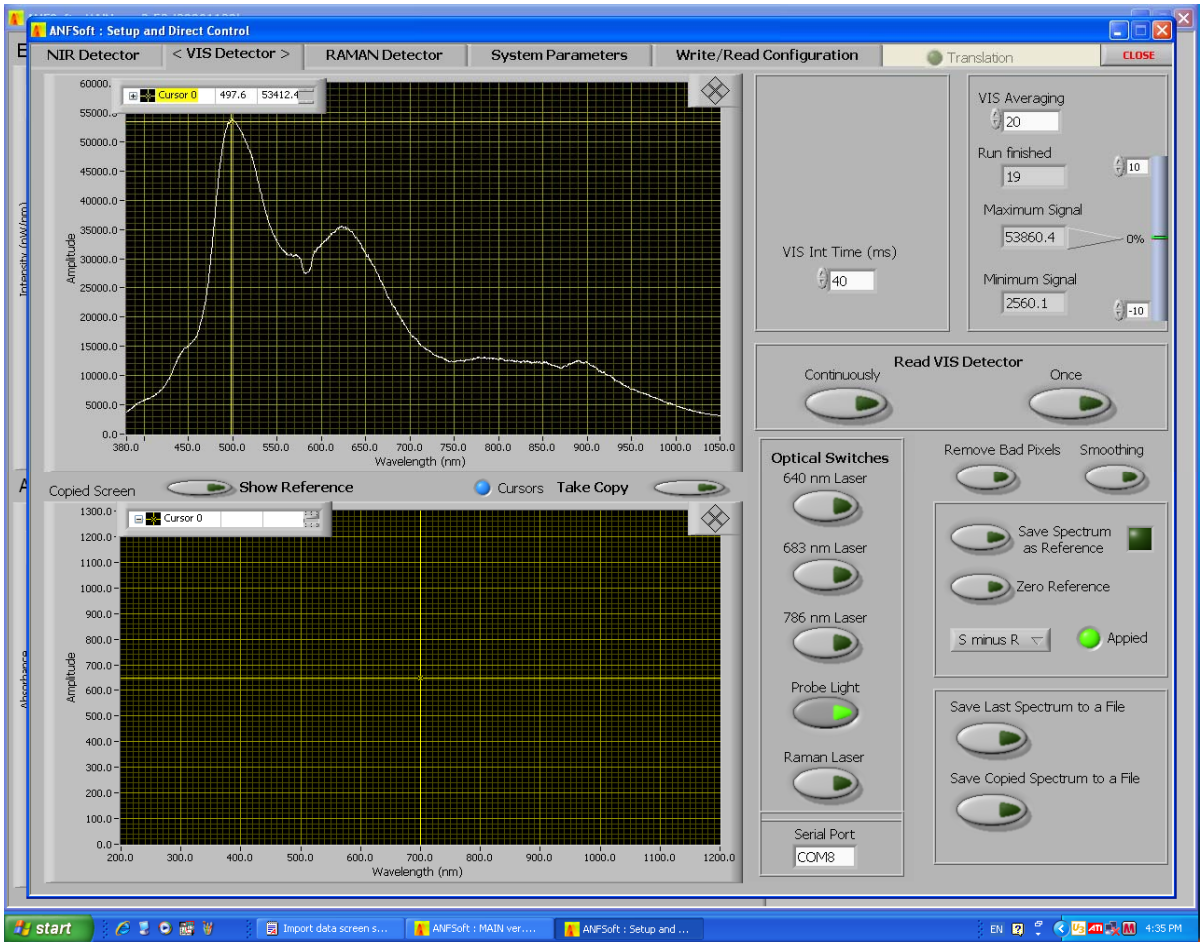


Fig. 19. The VIS Detector screen of Setup mode.

RAMAN DETECTOR TAB

The **RAMAN Detector** screen is shown below. It is very similar to the **VIS Detector** screen. Note that Raman spectra generally require much longer integrations than fluorescence or absorption spectra. The best signal-to-noise performance will be obtained by using long integration times with a few averages rather than shorter integration times and many averages. Because of hardware and software overhead, the total time between screen updates will be more than twice the value predicted from acquisition parameters.

The displayed Laser Wavelength is entered at the factory during calibration of the Raman shift frequency scale. Contact ANF if it appears that re-calibration is needed.

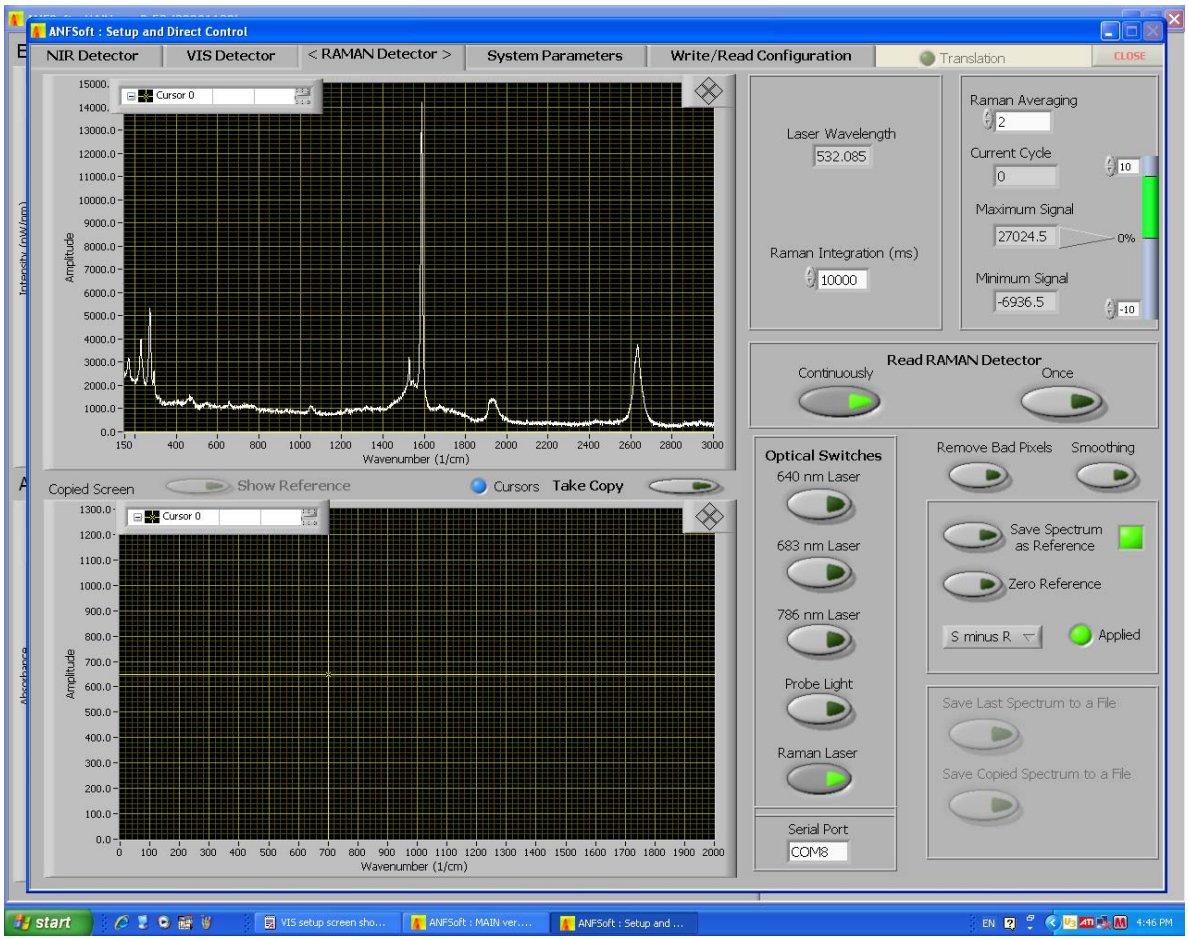


Fig. 20. The RAMAN Detector screen of Setup mode.

SYSTEM PARAMETERS TAB

The **Open Instrument Parameters Input** button is for maintenance functions not normally performed by end users.

Below and to the left of that button, the **Origin Window Limits** box allows the user to set the screen location and size of the Origin window. The **Graph Parameters** button opens a dialog box that allows selection of the graphs to be included in Origin project files. The user can specify inclusion of any combination of emission, absorption, and simulated excitation/emission contour with spectral axes in wavelength and/or wavenumber.

Below and to the right is a box that allows the user to specify the maximum idle time (in minutes) after which the tungsten probe lamp will be automatically turned off (to extend its life), and the minimum warm-up time (in seconds) after it is turned back on.

Just below that is the **Laser Delay and Sleep** group, containing separate warm-up delays (in seconds) for each laser before data acquisition. Delays may be useful for the highest data reproducibility when using the DPSS excitation lasers, but they are not needed for diode

lasers. To extend the service life of the DPSS lasers, enter a non-zero value (in minutes) in the corresponding **Sleep** fields. Then when a laser has not been used for the specified time, its emission will be paused. It will automatically restart when needed.

The lower right quadrant of the screen shows the serial numbers of the instrument's spectrometer and the identity of the computer COM port in use for instrument control. This information is displayed for maintenance purposes.

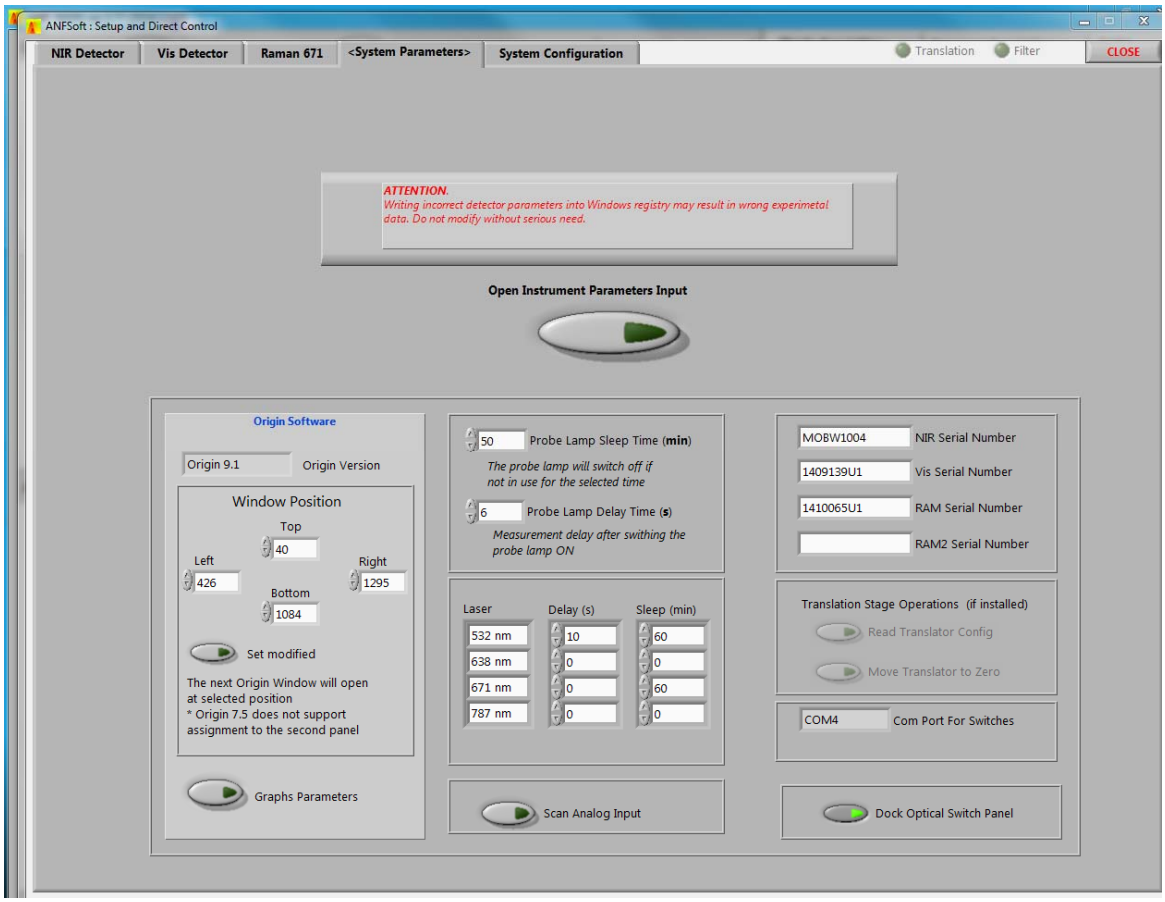


Fig. 21. The System Parameters screen of Setup mode.

WRITE / READ CONFIGURATION TAB

This screen provides a dialog box that allows one to save and recall full sets of experimental configuration parameters. This feature is very useful if the Instrument is shared among different projects or users. After setting up an optimized experimental configuration, a user can easily save that set of control and analysis parameters as a configuration file. That configuration can later be quickly reloaded using this dialog box

10. Troubleshooting

If the system will not start, the problem probably involves electrical connections or device drivers.

1. General information: In some cases, after a cable connection is changed, Windows may look in the wrong directory and not be able to automatically find some driver files. This happens most commonly with a change of USB port for the USB-to-serial converter device. All installed drivers (dll and sys files) are already present in the folder C:\WINDOWS\system32\drivers. To solve the problem, just point to this folder in the dialog box and proceed.
2. If the visible absorption option is installed and the visible detector does not respond during Software launch, an error message will appear before the colored "Nanoscape" icon. In this case the Software will not run and must be closed. After closing, check that the VIS detector is properly connected by its cable running from the back port of the Main Optical Unit to the computer's USB port. As of 2007, each serial-numbered NS2 instrument must have a driver named SM240-2 shown in the list of USB devices in the Windows Device Manager.
   a) If there is an exclamation mark in Device Manager and the driver was not installed, this is likely because driver re-installation was not finished after a USB cable relocation. When any USB device is connected to a new port never before used with that device, Windows reinstalls its driver. This process can be transparent, as for most USB storage devices, but for many other devices it will require user confirmation. If the visible detector's USB cable is reconnected to a different USB port that had never previously been used for the detector, a driver installation dialog box will appear. The user must confirm/accept the driver installation. Normally, Administrator rights on the computer are required for this process. Note that two drivers (one for the interface and another for the detector itself) will be sequentially installed for the visible detector, so do not stop the installation prematurely.
   b) If Device Manager shows no driver for the missing device, recheck the cable connection. Also, try to connect the visible detector cable to another USB port.
   c) After the SM240-2 device driver is installed, you may need to restart the NanoSpectralyze program before the detector can be used. (If there are problems closing the program in a normal way and it does not respond, use Task Manager.)
   d) If the above steps do not resolve a problem with the visible detector, contact ANF for advice or service.
3. The message "NIR detector did not respond…" may appear during the Software start. First, check that the power switch on the Spectrometer Module is ON. If this is not the problem, it is possible that the error message may appear even with a properly configured system. Click "Try again" to resolve the problem in this case. If this does not help and the message reappears, the main NIR detector is not responding correctly. Check that the MicronOSI driver is present in the Windows Device Manager.

a) If the device is not in the list, but there are uninstalled devices, try to install them, using a procedure similar to that described in the previous section.

b) If no MicronOSI device and no non-installed devices are displayed, check the USB cable connection between the computer and Spectrometer Module. Also check that the Spectrometer Module's power adapter is properly connected to the Module and to a working AC outlet, and that its green pilot light (if present) is lit.

c) When the MicronOSI detector connects and its driver installs, in most cases the NanoSpectralyze software should be able to proceed without restarting.

d) If an NIR spectrometer disconnection or power loss occurred while the ANF Software was running, it might continue normal operation when the connection is restored, without requiring a restart. However, if data acquisition was in process during the disconnection, the acquired data will be compromised and the experiment should be repeated.

e) If the above steps do not resolve a problem with the NIR detector, contact ANF for advice or service.

4. Another USB cable from the Main Optical Unit to the instrument computer is a control cable containing a USB-to-serial converter. Here are some communication-related events that may arise for this connection:

a) If the cable is moved to a different USB connector of the computer, Windows will assign it to a new COM port. This process may require the user to confirm installation of the cable (USB to serial converter) driver. This process will be transparent only if the computer's USB port had previously been used with the device and the driver had previously auto-configured for the port. At the next launch of the ANF Software after such a switch, NanoSpectralyze will inform the user about the COM port change. This is normal behavior and does not indicate a problem if the cable had been moved to another port.

b) The message "No power or connection to the main unit…" indicates a problem. Check that:

– the main unit is powered (green LED is lit) and the sample door is closed (the interlock status LED is lit)

– all connections within the USB-to-serial cable are secure

– the USB-to-serial converter driver is installed correctly (a corresponding device, usually called Prolific driver should be listed in the Device Manager in the USB controllers group). A proper connection will result in the appearance of associated with the device COM port, having a number 3 or higher in group Ports.

11. Maintenance

The NS2 optical system contains no components that are intended to be adjusted or serviced by the user. The enclosure of the Main Optical Module should therefore not be

removed. The sample door is electrically interlocked to prevent laser operation except when the door is fully closed. Do not attempt to defeat this interlock or to operate the NS2 with its enclosure removed, as such actions may expose users to hazardous laser beams.

The foam dust filter in the external fan guard of the Main Optical Module (on the side opposite the fiber optic cable port) should be periodically examined and washed or replaced when necessary. To remove this filter, simply pry off the central plastic section of the fan guard and lift out the foam filter sheet.

12. Applications Support

Applied NanoFluorescence staff is available to provide advice on the proper use of the NS2 hardware and the Software in your application. They may be contacted by e-mail at info@appliednano.com or by phone at 001-713-521-1450. Useful information is also posted in the Customer Support section of the ANF web site.

13. Warranty

Applied NanoFluorescence, LLC (ANF) warranties your NanoSpectralyzer for a period of one year following delivery. This warranty covers the parts and labor costs needed to repair defects or failures in materials and workmanship. If it appears that the unit needs repair, contact your distributor or ANF to describe the problem and arrange for service. If the service cannot be performed at your site, you will pay the return shipping costs and ANF will pay for the shipping back to you after repairs. This warranty will be void if the customer has removed the enclosure of or made modifications to any module without direct instructions from ANF.

In addition, ANF will provide updated versions of the standard NanoSpectralyze software at no charge for a period of three years following delivery. These software updates will be provided in forms intended for simple installation by end users, and will not include site visits for installation by ANF staff. See the Customer Support section of the ANF web site for software updates.

14. Disclaimer

Results obtained by the NanoSpectralyzer reflect the best efforts of ANF to characterize samples of single-walled carbon nanotubes on the basis of spectroscopic measurements and recent research findings in the field of nanotube spectroscopy. However, ANF will not be liable for any direct or consequential damages resulting from errors in data or deduced results obtained using the NanoSpectralyzer system.

Appendices

Appendix 1 – Specifications

NS2 S/N 031

Fluor. excitation source 1	115 mW at 532 nm (DPSS laser)
Fluor. excitation source 2	125 mW at 638 nm (diode laser)
Fluor. excitation source 3	35 mW at 671 nm (DPSS laser)
Fluor. excitation source 4	75 mW at 787nm (diode laser)
Fluorescence spectral range	882 –1579 nm
Absorption spectral range	410 –1579 nm
Near-IR detector type	TE-cooled 512-element InGaAs array
Near-IR detector temperature	-15 °C
Visible detector type	TE-cooled 2048-element CCD array (Sony)
Absorbance ceiling, near-IR	3.0 AU
Absorbance ceiling, visible	3.0 AU
Absorbance noise, near-IR	2×10^{-4} AU (rms) at 0 AU for 10 s integration
Absorbance noise, visible	5×10^{-4} AU (rms) at 0 AU for 10 s integration
Spectral resolution, near-IR	4 nm
Spectral resolution, visible	1.2 nm
SWCNT detectable fluor. diameter range	~ 0.7 - 1.4 nm
Raman excitation source	180 mW at 671 nm
Raman detector type	TE-cooled 2048-element CCD array (Sony)
Raman spectral resolution	5 cm^{-1}
Raman spectral range	150 - 3400 cm^{-1} shift
Raman S/N ratio	350 (CCl_4 459 cm^{-1} peak, 1 s integration)
Minimum sample volume	200 µL
Optic axis height in cell holder (Z-value)	8.5 mm above rubber pad (with shim removed)
Data acquisition time	~15 seconds per spectrum (typical)
Maximum spectral acquisition rate	>10 spectra per second (in sequence mode)
Main Optical Module dimensions	12.5" W x 18.5" D x 6.5" H (318 x 470 x 165 mm)
System weight	60 lbs (27 kg) excluding computer
Power consumption	250 watts (max), including computer and monitors

Appendix 2 – Certification

CE

EC Type Declaration of Conformity

We,

Applied NanoFluorescence, LLC

(Supplier's company name or representative in the EC)

3701 Kirby Drive
Suite 994
Houston, TX 77098 USA

(supplier's of representative's address)

declare under our sole responsibility that the product:

Model NS1 NanoSpectralyzer

name, type or model, batch or serial number, possibly source and number of items.

to which this declaration relates in conformity with the following European, harmonized and published standards at date of this declaration:

IEC 61010-1
IEC 61326, Edn. 1.0
IEC 60825-1

title and or number and year of issue of the applied standard(s)

following the provisions of the Directives (if applicable):

LVD directive 73/23/EEC and its amendment 93/68/EEC
EMC directive 89/336/EEC and its amendment 93/68/EEC

These conclusions were based on test report:
ANF lvd.2006.1. June 2006, ANF, LLC
ANF laser.2006.1 July 2006, ANF, LLC
Langston EMC.2006.1 July 2006, W. Langston, Inc.

test report number, date and name of test house and references to other documents

Houston, Texas, USA July 21, 2006

place and date of issue *last 2 digits of year the ce marking was affixed the first time*

R. Bruce Weisman

0	6

R. Bruce Weisman, President

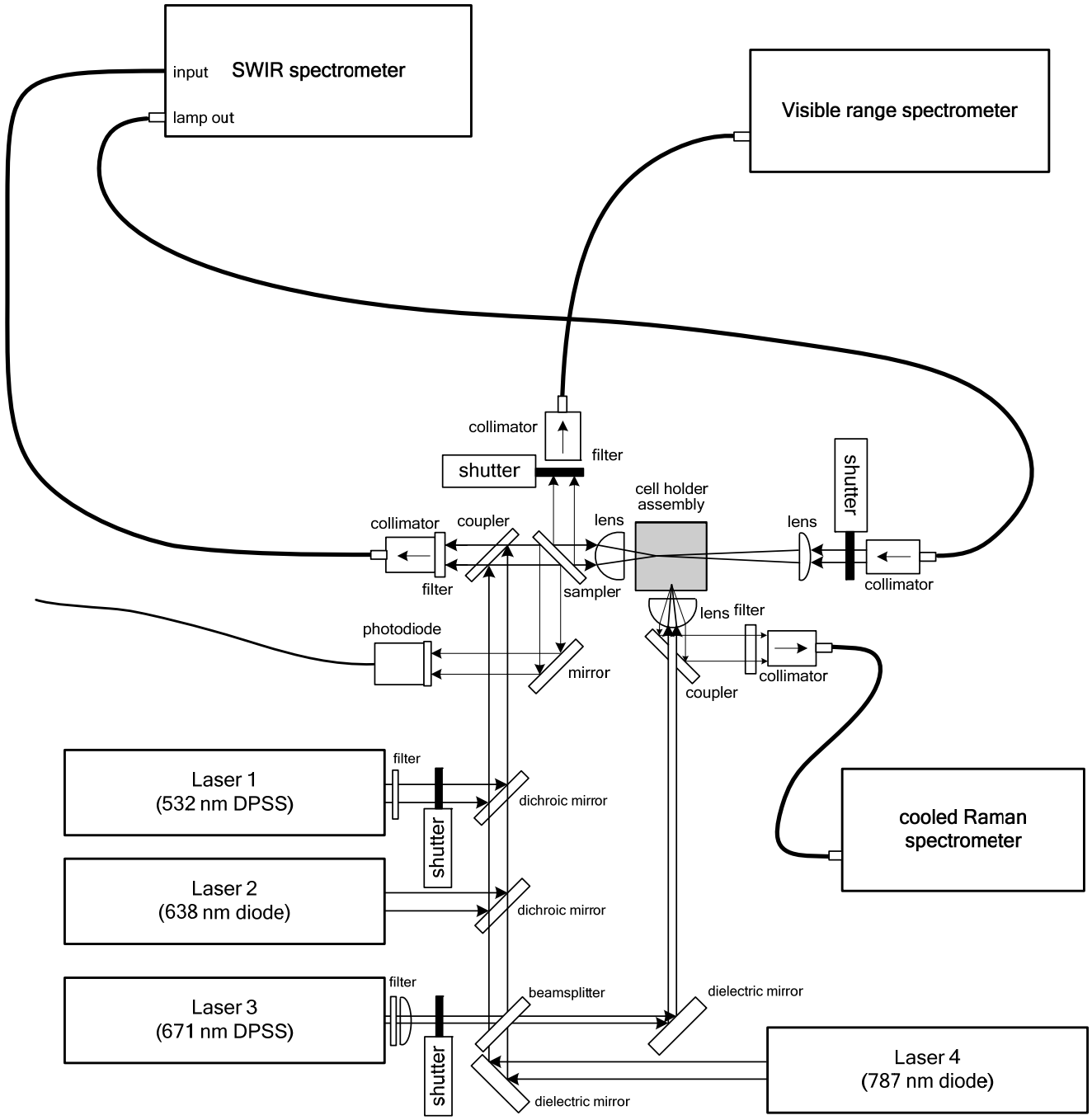
name and signature of company authorized person and companies stamp

This Declaration of Conformity is suitable to the European Standard EN 45014 *General Criteria for supplier's Declaration of Conformity.* The basis for the criteria has been found in international documentation, particularly in ISO/IEC, Guide 22, 1982, *Information on manufacturer's Declaration of Conformity with standards or other technical specifications.*

This declaration is a EC Type Declaration of Conformity as referenced in article 10.1 of EC directive 89/336/EEC *The EMC-directive* and as in Appendix III.B of EC directive 73/23/EEC *The Low Voltage Directive*

Appendix 3 – Optical Schematic

ANF Model NS2
(Unit 31, UQAM)



Appendix 4 – Performance Checks (Unit 31)

Fluorimetric Sensitivity

The fluorimetric sensitivity of the NS2 can be verified using the solid fluorescence reference standard (supplied). To check performance, perform the following steps:

1. With the cell holder empty, enter the **Setup** mode of the NanoSpectralyze software.
2. Set the, the gain to **High**, and deactivate the lamp.
3. Select **Remove Bad Pixels.**
4. Activate the first (638 nm) laser with a **NIR Int Time** of **100 ms**.
5. Select **Read Continuously**.
6. Select **Save Spectrum as Reference** and make sure the **Applied** button is lit.
7. Confirm a flat spectrum near zero with less than 25 units noise; deactivate the laser.
8. Place the solid reference standard (a 3 mm thick filter plate bonded to a clear spacer plate) into the cell holder. It needs to stand flush against the solid wall of the sample holder that is perpendicular to the long axis of the optical module. Be sure to orient the standard so that the clear glass spacer touches the sample holder wall.
9. Close the sample door, activate the laser, and note the peak signal at 1060 nm.
10. Deactivate the laser, remove the solid reference standard; close the sample door.
11. Activate the second (671 nm) laser and repeat the measurement (steps 5 through 10).
12. Activate the third (787 nm) laser and repeat the measurement (steps 5 through 10).
13. **Do not** repeat with the 532 nm (Raman) laser, because this beam will damage the solid fluorescence reference standard.
14. Return the solid fluorescence reference standard to a safe long-term storage location.

The nominal peak intensity of the 1060 nm emission peak for your system is:

4900 units with 638 nm excitation, in 100 ms

3900 units with 671 nm excitation, in 100 ms

42,000 units with 787 nm excitation, in 100 ms

(do not measure) with 532 nm excitation

If your system gives intensity readings more than 15% below these values, please contact ANF.

Raman Sensitivity

The Raman sensitivity of the NS2 can be verified using a liquid sample cell containing pure carbon tetrachloride (CCl_4):

1. With the cell holder empty, enter the **Setup** mode of the NanoSpectralyze software and select the **Raman** tab.
2. Enter 1000 ms for **Raman integration** time and 5 for **Raman averaging**.
3. Select the **Raman Laser** to activate it
4. Select **Continuously** to acquire a blank spectrum
5. Select **Save Spectrum as Reference** and make sure the adjacent square green indicator is lit.
6. Deactivate the **Raman Laser**
7. Open the sample door and place a liquid cell containing carbon tetrachloride into the cell holder
8. Close the sample door and activate the **Raman Laser**.
9. After a stable Raman spectrum is displayed, stop data acquisition by clicking the **Continuously** button.
10. Note the magnitude of the Raman peak at 459 cm^{-1}.

The nominal peak magnitude for your system is:

__8500_ units

If you observe intensity readings more than 15% below this value, please contact ANF.

Appendix 5 – Global Fit Parameters and Guidelines

A general approach to spectral fitting with a new sample is first to load the template intended for or most similar to that sample's surfactant. A **Quick Fit** should be run to see how accurate a simulation is obtained with the default parameter values of that template. If improvements are needed, a **Full Fit** analysis should be run with some of the above parameters allowed to vary. As the fit quality improves, fix some of the parameters that had been varied and allow new ones to vary. Some iteration of this procedure may be needed, but it is generally possible to reduce the MSD value to ~0.005. Once a satisfactory high quality fit has been obtained, save the parameters as a new template that will reflect the environment and history of the analyzed sample. Subsequent analyses of similarly prepared samples can then be very quickly computed using this template, with little or no need for parameter variation.

In the following paragraphs, the meaning of each fit parameter is described along with its recommended variation limit. Note that these limits apply to a single round of fitting. The fitting process can be restarted from the last set of parameter values by select **Calculate** again on the Global Fit screen. In these subsequent fitting rounds, parameters are again freed to vary by the range specified in the **Variation %** fields. Therefore running five consecutive fitting rounds with a 10% allowed variation in some parameter can lead to a much larger cumulative variation of ~50%. Keep this effect in mind when setting variation limits. Note also that the fitting routine searches for an optimal solution in a multidimensional parameter space. Decreasing the number of varied parameters will therefore greatly accelerate convergence. Decreasing the range of allowed variation for those parameters that are known relatively precisely will also speed the fitting process.

- **E11 Position** is the set of (*n*,*m*)-specific peak frequencies, in cm^{-1}, for the emission features (first van Hove transitions). The array size is equal to the number of SWNT types used in the fitting, the number is the same as for (see below) **E22 Position**, **E11 Width**, **E22 Width**, **E11 Shape**, **E11 Exc Factor**, and **Solution Vector**, all these are parameters, independently variable for every SWNT type. These values can vary with SWNT quality, the surfactant or other environment conditions and, sometimes, they depend on sample preparation protocol used. When using a set of frequencies that is appropriate for the relevant surfactant, only slight variations (within ~0.05-0.1%) should be permitted for **E11 Position**. Larger tolerances may lead to fits that appear accurate but involve misidentification of some peaks.
- **E22 Position** is the set of (*n*,*m*)-specific peak frequencies, in cm^{-1}, for the absorption features (second van Hove transitions). These values are somewhat less critical than the E11 positions and may be safely allowed to vary by ~0.1%.
- **E11 Width** is the set of (*n*,*m*)-specific individual peak widths (FWHM, in cm^{-1}) for the emission features. During initial fitting of a sample without a refined template, it is recommended to use the **Shared E11 Width** parameter instead. This will prevent individual peak widths from assuming anomalously small or large values. After a rather refined fit has been obtained with **Shared E11 Width**, spectral fitting can proceed with individual width variations using the E11 Width parameters. Here, variations of ~3% per

round may be safely allowed, but after a few rounds the values should be checked and corrected to avoid unreasonable broadening (most frequent problem) or narrowing of the component peaks. Examination of peak contours in the Simulated Contour Plot display (see Fig. 16) can help to identify such problems.

- **E22 Width** is the set of (*n*,*m*)-specific peak widths (FWHM, in cm^{-1}) for the absorption features. It is similar to **E11 Width**.
- **E11 Shape** is a set of (*n*,*m*)-specific ratio of Lorentzian to Gaussian characters (widths) used in convolution-based Voigt profiles modeling the emission components. The value changes from 0 to 1 upon transition from Gaussian to Lorentzian shape. It is possible to allow variations of ~10% in these parameters, but a smaller number will accelerate the search for other parameters. Manual adjustment of these values after a few calculation rounds may be required to avoid "sticking" at unrealistic values of 0 or 1. Fitting more frequently will tend to overestimate the parameter, since Lorentzian profile has longer wings.
- **E11 Exc Factor** is a set of parameters that accounts for the possible excitation of E_{11} vibronic sidebands by the lasers. Variations of ~25% may be allowed. In the present version one set of four values is used for all SWNT types, representing the first, second, and third vibronic (exciton-phonon) features to the high-frequency side of the main excitation peak, plus a constant background. The parameters should be kept relatively small, with the first one below 0.2.
- **Shared E11 Width** is a single peak width (FWHM, in cm^{-1}) used to describe all of the emission features (as an alternative to (*n*,*m*)-specific peak widths). Selecting a variation in this parameter disables variations of the **E11 Widt**h parameter set described above. A 20% variation range can be allowed in exploratory fitting, but the parameter can be fixed for subsequent analyses of similarly prepared samples.
- **Shared E11 Shape** is a single ratio of Lorentzian to Gaussian character used in modeling all of the peaks in the emission spectrum (as an alternative to (*n*,*m*)-specific peak shapes). Selecting a variation in this parameter disables variations of the **E11 Shape** parameter set described above. As for the shared E11 width, this parameter can be varied rather widely during exploratory fitting but should then be stable within sets of similarly prepared samples.
- **Excitation Profile** is a set of common parameters used in modeling the vibronic structure of the E_{22} absorption features. These parameters can be varied by ~10% during exploratory fitting but should then be stable within sets of similarly prepared samples.
- **Laser Ratio** holds an array of N_L parameters (where N_L is the number of excitation lasers) describing the relative laser powers and optical efficiencies at the excitation wavelengths. After fitting the first value is equal to 1. Once optimized, this experimental parameter should remain stable for long periods, since it represents a real ratio of laser intensities corrected slightly for excitation efficiency. However, when relatively concentrated samples or samples of non-standard geometry are analyzed, the parameter may need adjustment to account for differing penetration depths of the excitation beams in the

sample cell. A variation limit of ~5% per fitting round is recommended using an established template, but a limit of 20% or higher can be used for a new template when the array begins with a set of values equal to 1.

- **Solution Vector** shows the set of deduced (*n,m*) concentrations found through the fitting process. These are the values transmitted to Origin and used to prepare the displayed results graphs and worksheets.

Appendix 6 – Structure of the Origin Template and Project Files

The Origin template files are used to define the structure of the project files. Project files are created from template files by populating the template worksheets with data and generating plots from the worksheets. By using the Global Fit Parameters Dialog, any compatible template or pre-existing project file can be imported as a template file. New template or project files are created starting with the Global Fit Default.opj file by modifying data in the worksheets and, for projects, adding plots. From the Global Fit screen, **Save as Template** creates a small template file with current fit parameters but no data.

Each project file includes the following graphs:

- **AbsCombined** shows the combined visible and near-IR absorbance spectra of the sample.
- **Fluor**nnn shows the emission spectrum with computed fit, where "nnn" is the wavelength in nanometers of the corresponding excitation laser. Prominent peaks are labeled with their dominant (*n,m*) components near the top of the graph.
- **Raman_Plot** shows the Raman spectrum of the sample, in units of counts per second.
- **ProfileSWNT** shows a color-coded two-dimensional contour plot of emission intensity as a function of excitation wavelength and emission wavelength, simulating data from a full excitation-emission scan. One-dimensional profiles of emission intensity as a function of each of these variables are plotted next to the contour plot. The wavelengths selected for the profile plotting may be changed by dragging the cross lines with the mouse.

> **Important Note**
>
> Because the NS2 measures emission profiles only for four specific excitation wavelengths, this contour plot is not directly measured. Instead, it is synthesized from the (*n,m*) distribution deduced by analyzing the four emission spectra. The appearance and accuracy of this contour plot therefore depend on the parameters found in the fitting routine. Inaccurate or anomalous fit parameters may give unrealistic contour plots. Please do not report such contour plots without describing them as synthesized.

- **Graphene_sheet** shows a graphene sheet plot of the relative abundances of various semiconducting (*n,m*) species in the sample. The thickness of the hexagonal border surrounding an (*n,m*) label is proportional to the deduced relative abundance.

- **Graph_COR** shows the graphene sheet plot after abundances have been corrected by a set of (*n*,*m*)-dependent fluorimetric sensitivity factors. These calibration factors are based on the latest SWCNT research, and are found in the Origin project file "Sensitivity.opj."
- **Histogram** shows a column histogram graph of the deduced diameter distribution of semiconducting nanotubes in the sample. The mean and standard deviation of the distribution are also shown.
- **HgramCOR** shows the histogram graph corresponding to **Graph_COR**, incorporating the fluorimetric calibration factors in "Sensitivity.opj".

In addition, each project file includes the following worksheets:

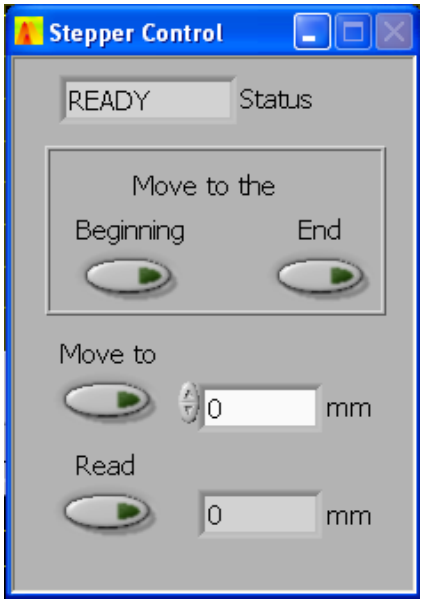
- **DATA1** contains the final values of all parameters used in the global fitting process. Most columns are easily identified from their labels. The one marked "exc" has (*n*,*m*)-dependent excitation probabilities, and "int" has the integrated intensities deduced in the fitting process.
- **DATA**nnn contains emission spectra expressed in several different units, plus the raw ratio of fluorescence to absorbance as a function of wavelength.
- **FIT**nnn contains the simulated emission spectrum plus individual (*n*,*m*) component spectra as plotted in **Fluor**nnn.
- **GRAPHENEDATA** contains the data used to generate the **Graphene_sheet** plot.
- **HISTDATA** contains the data used to construct the diameter **Histogram** plot.
- **INDEX**nnn contains data used to construct the (*n*,*m*) peak labels on the **Fluor**nnn plots.
- **NIR** contains the near-IR absorbance spectral data.
- **RESULTSGF** contains tabulated results from the global fitting computation.
- **VIS** contains the visible absorbance spectral data.

Appendix 7 – Vertical Translation Option (VTO)

In units equipped with the vertical translation option (VTO), the sample cell holder is mounted on a vertical translation stage driven by a computer-controlled stepper motor. This allows automatic scans of fluorescence, absorption, and Raman spectra as a function of position in the sample cell, using the Sequence Acquisition tab of the Main control screen. Such spectral maps are particularly valuable for *in situ* characterization of layered contents in centrifuge tubes used for density gradient ultracentrifugation sorting of SWCNT samples.

The VTO cell holder is designed to accommodate centrifuge tubes with outer dimensions of 13 x 51 mm or 14 x 89 mm (diameter x height), in addition to conventional square 12.5 x 12.5 mm spectrophotometer cells. When using square spectrophotometer cells or the shorter centrifuge tubes, it is necessary to place the cylindrical metal spacer at the bottom of the cell holder to raise the sample to an appropriate height. This spacer is made of magnetic stainless steel so that it can be easily inserted and removed using the magnetic pickup tool that is also provided with the instrument. The spacer should not be used with 14 x 89 mm centrifuge tubes.

On startup, the NanoSpectralyze program automatically moves the sample holder to its lowest position (designated 0 mm). The dialog box (shown below) displays the current height of the sample holder and allows user positioning to any height between 0 and 50 mm. A setting near 22 mm is recommended when using conventional square sample cells. This places the optical interrogation region within approximately 3 mm of the cell bottom, allowing accurate measurements with minimal sample volumes.

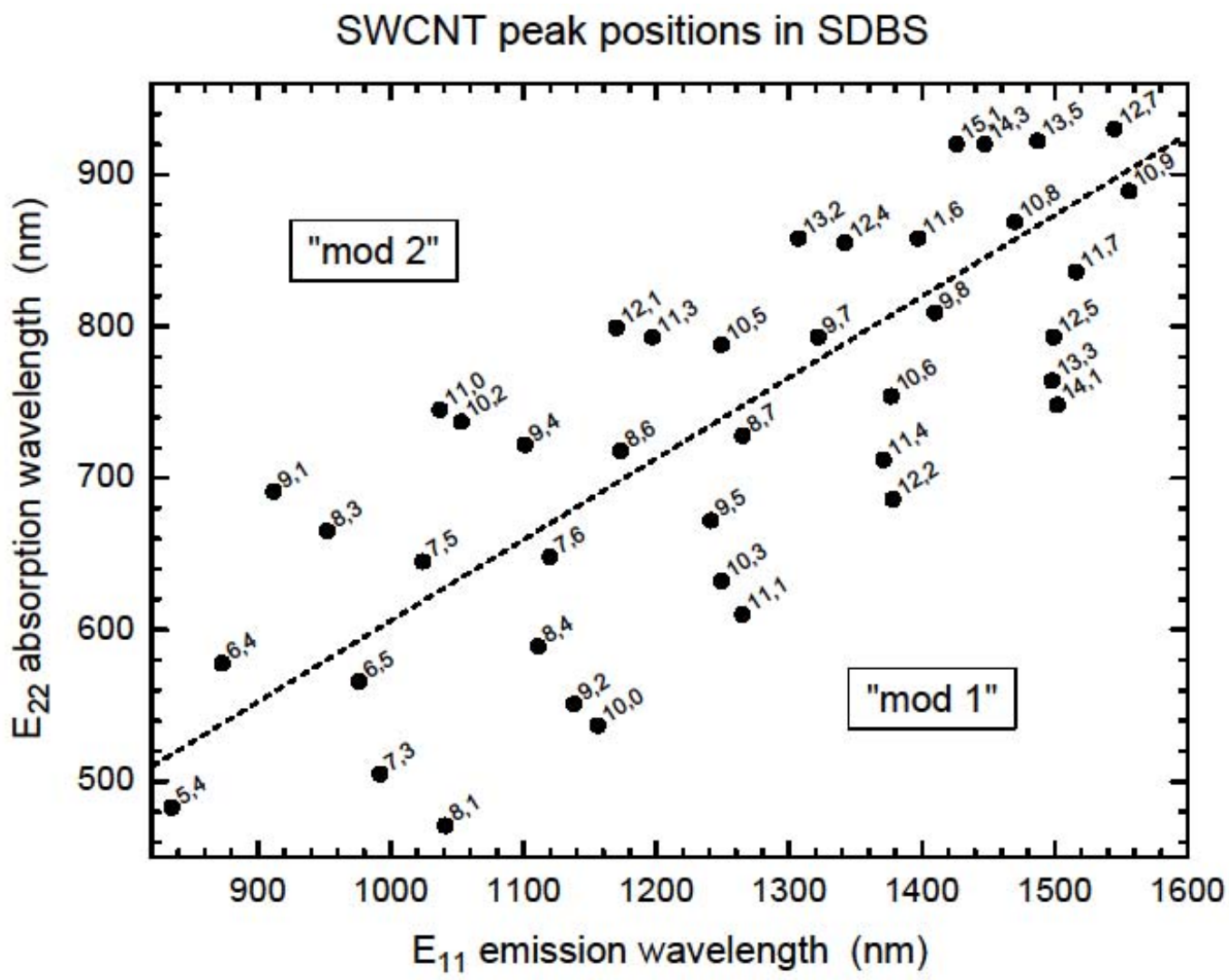


Appendix 8 – SWCNT Spectral Data and Spectral Map (in SDS or SDBS)

Table 1. Structures and First and Second van Hove Optical Transitions[a] for Semiconducting SWNT Structures with Diameters between 0.48 and 2.0 nm

(n, m)	d_t (nm)	α (deg)	mod[b]	λ_{11} (nm)	$\bar{\nu}_{11}$ (cm^{-1})	E_{11} (eV)[c]	λ_{22} (nm)	$\bar{\nu}_{22}$ (cm^{-1})	E_{22} (eV)
(4, 3)	0.483	25.28	1	700	14 283	1.771	398	25 147	3.118
(5, 3)	0.556	21.79	2	720	13 884	1.721	522	19 147	2.374
(5, 4)	0.620	26.33	1	835	11 974	1.485	483	20 697	2.566
(6, 1)	0.521	7.59	2	653	15 323	1.900	632	15 828	1.962
(6, 2)	0.572	13.90	1	894	11 183	1.387	418	23 900	2.963
(6, 4)	0.692	23.41	2	873	11 452	1.420	578	17 312	2.146
(6, 5)	0.757	27.00	1	976	10 244	1.270	566	17 667	2.190
(7, 0)	0.556	0.00	1	962	10 397	1.289	395	25 318	3.139
(7, 2)	0.650	12.22	2	802	12 468	1.546	626	15 977	1.981
(7, 3)	0.706	17.00	1	992	10 083	1.250	505	19 820	2.457
(7, 5)	0.829	24.50	2	1024	9768	1.211	645	15 496	1.921
(7, 6)	0.895	27.46	1	1120	8930	1.107	648	15 441	1.914
(8, 0)	0.635	0.00	2	776	12 886	1.598	660	15 146	1.878
(8, 1)	0.678	5.82	1	1041	9603	1.191	471	21 226	2.632
(8, 3)	0.782	15.30	2	952	10 508	1.303	665	15 029	1.863
(8, 4)	0.840	19.11	1	1111	8997	1.116	589	16 981	2.105
(8, 6)	0.966	25.28	2	1173	8525	1.057	718	13 928	1.727
(8, 7)	1.032	27.80	1	1265	7908	0.981	728	13 727	1.702
(9, 1)	0.757	5.21	2	912	10 964	1.359	691	14 466	1.794
(9, 2)	0.806	9.83	1	1138	8790	1.090	551	18 155	2.251
(9, 4)	0.916	17.48	2	1101	9086	1.126	722	13 843	1.716
(9, 5)	0.976	20.63	1	1241	8055	0.999	672	14 883	1.845
(9, 7)	1.103	25.87	2	1322	7567	0.938	793	12 610	1.563
(9, 8)	1.170	28.05	1	1410	7093	0.879	809	12 362	1.533
(10, 0)	0.794	0.00	1	1156	8652	1.073	537	18 606	2.307
(10, 2)	0.884	8.95	2	1053	9493	1.177	737	13 574	1.683
(10, 3)	0.936	12.73	1	1249	8006	0.993	632	15 834	1.963
(10, 5)	1.050	19.11	2	1249	8006	0.993	788	12 695	1.574
(10, 6)	1.111	21.79	1	1377	7262	0.900	754	13 262	1.644
(10, 8)	1.240	26.33	2	1470	6805	0.844	869	11 502	1.426
(10, 9)	1.307	28.26	1	1556	6428	0.797	889	11 248	1.395
(11, 0)	0.873	0.00	2	1037	9644	1.196	745	13 431	1.665
(11, 1)	0.916	4.31	1	1265	7906	0.980	610	16 388	2.032
(11, 3)	1.014	11.74	2	1197	8353	1.036	793	12 617	1.564
(11, 4)	1.068	14.92	1	1371	7295	0.904	712	14 036	1.740
(11, 6)	1.186	20.36	2	1397	7157	0.887	858	11 661	1.446
(11, 7)	1.248	22.69	1	1516	6597	0.818	836	11 968	1.484
(11, 9)	1.377	26.70	2	1617	6183	0.767	947	10 564	1.310
(11, 10)	1.444	28.43	1	1702	5877	0.729	969	10 320	1.280
(12, 1)	0.995	3.96	2	1170	8549	1.060	799	12 516	1.552
(12, 2)	1.041	7.59	1	1378	7255	0.900	686	14 575	1.807
(12, 4)	1.145	13.90	2	1342	7452	0.924	855	11 693	1.450
(12, 5)	1.201	16.63	1	1499	6670	0.827	793	12 605	1.563
(12, 7)	1.321	21.36	2	1545	6473	0.803	930	10 751	1.333
(12, 8)	1.384	23.41	1	1657	6036	0.748	917	10 910	1.353
(12, 10)	1.515	27.00	2	1765	5666	0.703	1024	9763	1.210
(12, 11)	1.582	28.56	1	1848	5412	0.671	1049	9535	1.182
(13, 0)	1.032	0.00	1	1384	7228	0.896	677	14 770	1.831
(13, 2)	1.120	7.05	2	1307	7651	0.949	858	11 661	1.446
(13, 3)	1.170	10.16	1	1498	6676	0.828	764	13 095	1.624
(13, 5)	1.278	15.61	2	1487	6723	0.834	922	10 843	1.344
(13, 6)	1.336	17.99	1	1632	6127	0.760	874	11 441	1.419
(13, 8)	1.457	22.17	2	1692	5909	0.733	1004	9956	1.234
(13, 9)	1.521	24.01	1	1799	5558	0.689	997	10 027	1.243
(13, 11)	1.652	27.25	2	1912	5230	0.648	1102	9071	1.125
(13, 12)	1.719	28.68	1	1994	5015	0.622	1128	8862	1.099
(14, 0)	1.111	0.00	2	1295	7721	0.957	859	11 640	1.443
(14, 1)	1.153	3.42	1	1502	6660	0.826	748	13 364	1.657
(14, 3)	1.248	9.52	2	1447	6910	0.857	920	10 867	1.347
(14, 4)	1.300	12.22	1	1623	6162	0.764	842	11 875	1.472
(14, 6)	1.411	17.00	2	1633	6123	0.759	992	10 078	1.250
(14, 7)	1.470	19.11	1	1768	5655	0.701	955	10 476	1.299
(14, 9)	1.594	22.85	2	1840	5435	0.674	1080	9261	1.148
(14, 10)	1.658	24.50	1	1942	5148	0.638	1078	9279	1.150
(14, 12)	1.789	27.46	2	2059	4856	0.602	1181	8470	1.050
(14, 13)	1.857	28.78	1	2141	4672	0.579	1208	8278	1.026
(15, 1)	1.232	3.20	2	1426	7011	0.869	920	10 864	1.347
(15, 2)	1.278	6.18	1	1622	6165	0.764	822	12 163	1.508
(15, 4)	1.377	11.52	2	1589	6294	0.780	986	10 140	1.257
(15, 5)	1.431	13.90	1	1752	5708	0.708	921	10 858	1.346
(15, 7)	1.546	18.14	2	1779	5621	0.697	1064	9396	1.165
(15, 8)	1.606	20.03	1	1907	5245	0.650	1035	9663	1.198
(15, 10)	1.730	23.41	2	1987	5032	0.624	1156	8650	1.072
(15, 11)	1.795	24.92	1	2086	4793	0.594	1158	8635	1.071
(15, 13)	1.927	27.64	2	2207	4532	0.562	1259	7942	0.985
(15, 14)	1.994	28.86	1	2287	4373	0.542	1288	7767	0.963
(16, 0)	1.270	0.00	1	1623	6163	0.764	815	12 264	1.521
(16, 2)	1.357	5.82	2	1561	6405	0.794	984	10 162	1.260
(16, 3)	1.405	8.44	1	1746	5728	0.710	898	11 139	1.381
(16, 5)	1.508	13.17	2	1732	5775	0.716	1055	9480	1.175
(16, 6)	1.564	15.30	1	1884	5307	0.658	1000	9999	1.240
(16, 8)	1.680	19.11	2	1925	5194	0.644	1138	8788	1.090
(16, 9)	1.741	20.82	1	2046	4887	0.606	1115	8968	1.112
(16, 11)	1.867	23.90	2	2134	4685	0.581	1233	8111	1.006
(16, 12)	1.932	25.28	1	2231	4483	0.556	1238	8077	1.001
(17, 0)	1.350	0.00	2	1552	6443	0.799	984	10 167	1.261
(17, 1)	1.391	2.83	1	1744	5733	0.711	886	11 289	1.400
(17, 3)	1.483	7.99	2	1699	5886	0.730	1050	9525	1.181
(17, 4)	1.533	10.33	1	1873	5340	0.662	974	10 263	1.272
(17, 6)	1.641	14.56	2	1875	5332	0.661	1125	8886	1.102
(17, 7)	1.697	16.47	1	2019	4952	0.614	1079	9264	1.149
(17, 9)	1.816	19.93	2	2072	4827	0.599	1212	8248	1.023
(17, 10)	1.877	21.49	1	2188	4571	0.567	1195	8367	1.037
(18, 1)	1.470	2.68	2	1682	5944	0.737	1048	9543	1.183
(18, 2)	1.515	5.21	1	1868	5353	0.664	958	10 433	1.294
(18, 4)	1.611	9.83	2	1838	5439	0.674	1118	8947	1.109
(18, 5)	1.663	11.93	1	2003	4993	0.619	1052	9508	1.179
(18, 7)	1.773	15.75	2	2020	4952	0.614	1197	8351	1.035
(18, 8)	1.831	17.48	1	2156	4638	0.575	1159	8629	1.070
(18, 10)	1.951	20.63	2	2218	4509	0.559	1288	7765	0.963
(19, 0)	1.508	0.00	1	1867	5357	0.664	953	10 492	1.301
(19, 2)	1.594	4.95	2	1816	5507	0.683	1114	8979	1.113
(19, 3)	1.641	7.22	1	1994	5015	0.622	1033	9683	1.201
(19, 5)	1.741	11.39	2	1979	5052	0.626	1187	8422	1.044
(19, 6)	1.795	13.29	1	2135	4684	0.581	1130	8853	1.098
(19, 8)	1.907	16.76	2	2164	4621	0.573	1271	7869	0.976
(19, 9)	1.966	18.35	1	2295	4358	0.540	1238	8076	1.001
(20, 0)	1.588	0.00	2	1808	5531	0.686	1113	8989	1.114
(20, 1)	1.629	2.42	1	1990	5024	0.623	1023	9775	1.212
(20, 3)	1.719	6.89	2	1952	5124	0.635	1181	8466	1.050
(20, 4)	1.768	8.95	1	2122	4712	0.584	1108	9025	1.119
(20, 6)	1.872	12.73	2	2121	4715	0.585	1258	7947	0.985
(20, 7)	1.927	14.46	1	2269	4406	0.546	1208	8280	1.027
(21, 1)	1.708	2.31	2	1938	5161	0.640	1178	8487	1.052
(21, 2)	1.752	4.50	1	2116	4727	0.586	1095	9134	1.132
(21, 4)	1.847	8.57	2	2090	4786	0.593	1250	8000	0.992
(21, 5)	1.897	10.44	1	2253	4439	0.550	1184	8445	1.047
(22, 0)	1.747	0.00	1	2114	4731	0.587	1090	9171	1.137
(22, 2)	1.831	4.31	2	2070	4830	0.599	1245	8030	0.996
(22, 3)	1.877	6.31	1	2243	4459	0.553	1168	8560	1.061
(22, 5)	1.975	10.02	2	2229	4487	0.556	1320	7575	0.939
(23, 0)	1.826	0.00	2	2064	4845	0.601	1244	8039	0.997
(23, 1)	1.867	2.11	1	2238	4467	0.554	1160	8620	1.069
(23, 3)	1.956	6.05	2	2205	4535	0.562	1314	7612	0.944
(24, 1)	1.946	2.02	2	2193	4560	0.565	1311	7631	0.946
(24, 2)	1.990	3.96	1	2365	4229	0.524	1231	8121	1.007
(25, 0)	1.985	0.00	1	2362	4233	0.525	1228	8146	1.010

[a] As predicted by the empirical formulas in the text. [b] The value of mod($n - m$, 3). [c] E_{11} values represent emission peaks; to obtain absorption peak energies, add ca. 0.004 eV.


SWCNT peak positions in SDBS
"mod 2"
"mod 1"
E_{22} absorption wavelength (nm)
E_{11} emission wavelength (nm)
900
800
700
600
500
900
1000
1100
1200
1300
1400
1500
1600
15,1
14,3
13,5
12,7
10,9
13,2
12,4
11,6
10,8
11,7
12,1
11,3
10,5
9,7
9,8
12,5
11,0
10,2
9,4
8,6
8,7
10,6
13,3
14,1
11,4
9,1
12,2
8,3
9,5
7,5
7,6
10,3
11,1
6,4
8,4
6,5
9,2
10,0
7,3
5,4
8,1