Applied NanoFluorescence

Model NS3 NanoSpectralyzers

Training PowerPoint
Applied NanoFluorescence, LLC
2018

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What are NanoSpectralyzers?

Versatile multi-mode spectrometers optimized for nanomaterial characterization designed by experts in nanotechnology for scientific research.

NS1 NS2 NS3

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What makes it unique?

Able to measure a large variety of nanomaterials through their optical properties

Specializes in near-infrared (NIR) fluorescence spectroscopy

Patented software created to analyze single-walled carbon nanotubes and deduce a samples diameter and (*n*,*m*) distribution

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NanoSpectralyzers: Model Overview

NS1 NanoSpectralyzer

includes 4 excitation lasers; 900-1600 nm range for fluorescence; 410-1600 nm range for absorption

NS2 NanoSpectralyzer

includes all functions of NS1 plus Raman spectroscopy with choice of 532 nm or 671 nm excitation

NS3 NanoSpectralyzer

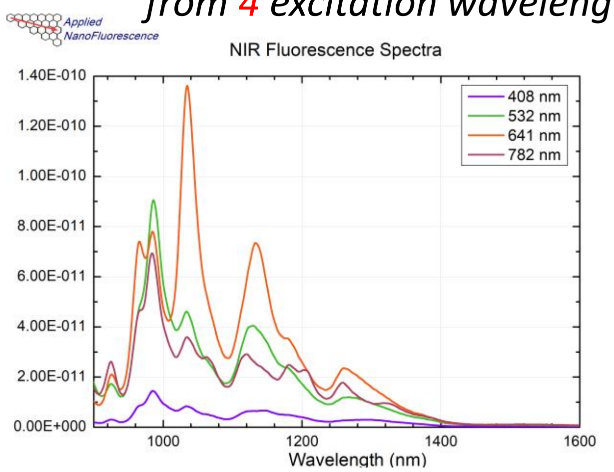
modular, customizable multi-mode system with 5 lasers for versatile nanomaterial characterization

NS MiniTracer (New!)

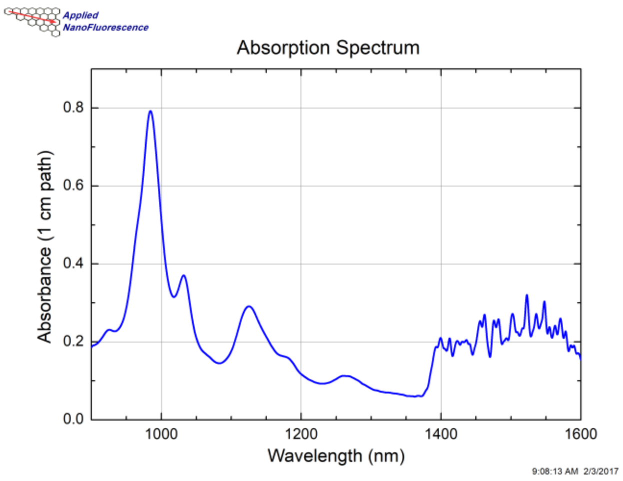
includes 1 excitation laser; 900-1600 nm range for fluorescence; optional NIR absorption

NS1 Spectroscopy Modules

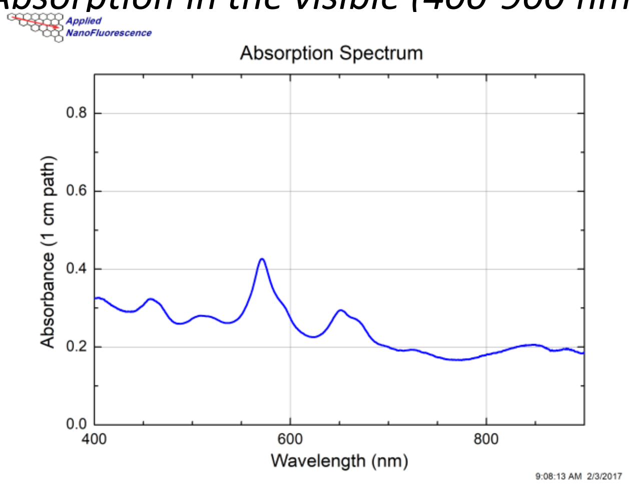
Fluorescence in the near-infrared (900-1600 nm) from 4 excitation wavelengths



Absorption in the near-infrared (900-1600 nm)



Absorption in the visible (400-900 nm)



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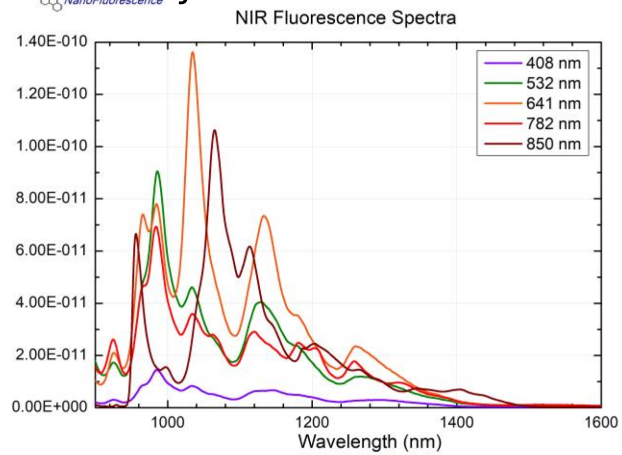
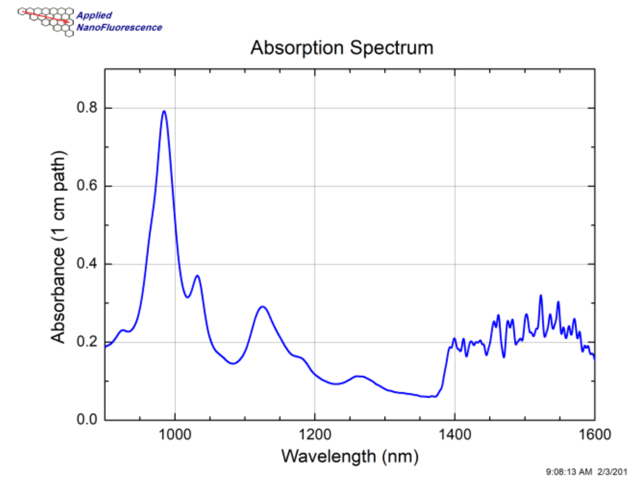
NS2 Spectroscopy Modules

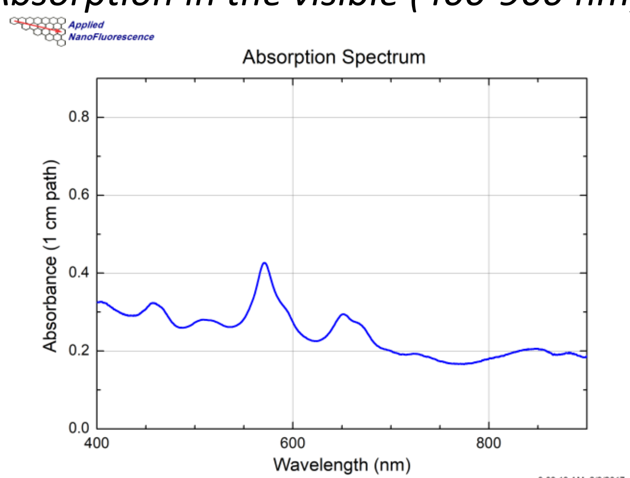
Fluorescence in the near-infrared (900-1600 nm) from 5 excitation wavelengths



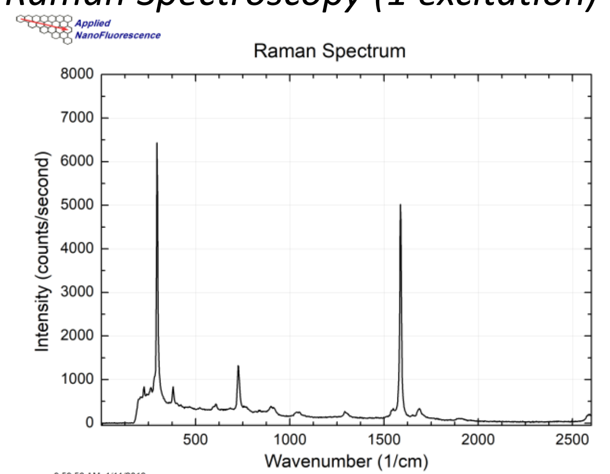
Absorption in the near-infrared (900-1600 nm)



Absorption in the visible (400-900 nm)



Raman Spectroscopy (1 excitation)



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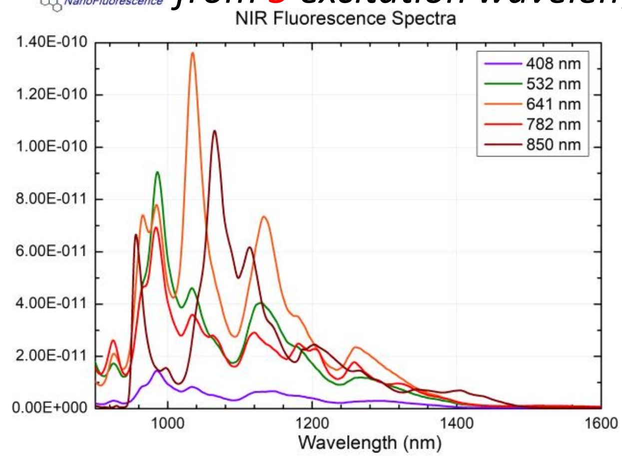
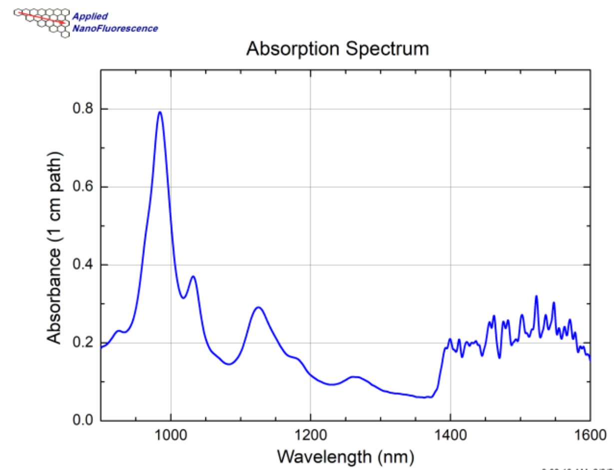
NS3 Spectroscopy Modules

Fluorescence in the near-infrared (900-1600 nm) from 5 excitation wavelengths



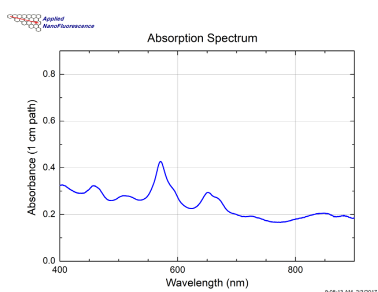
Absorption in the near-infrared (900-1600 nm)



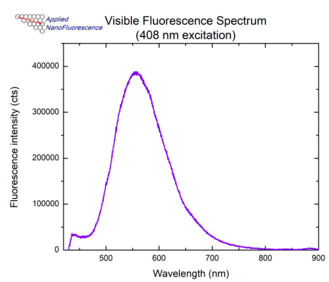
Included in Base System

Optional Modules

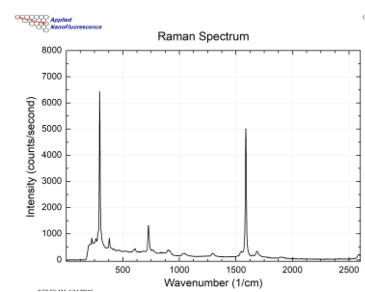
Absorption in the visible (400-900 nm)

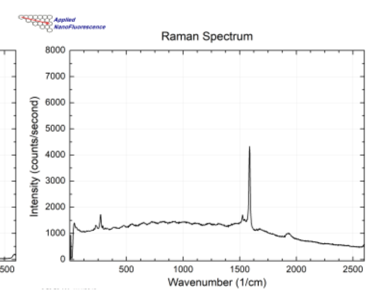


Fluorescence in the visible (450-900 nm) with same 5 excitations



Raman Spectroscopy (1 or 2 excitations)





UV absorption also available

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What spectroscopy modules are available?

Fluorescence:

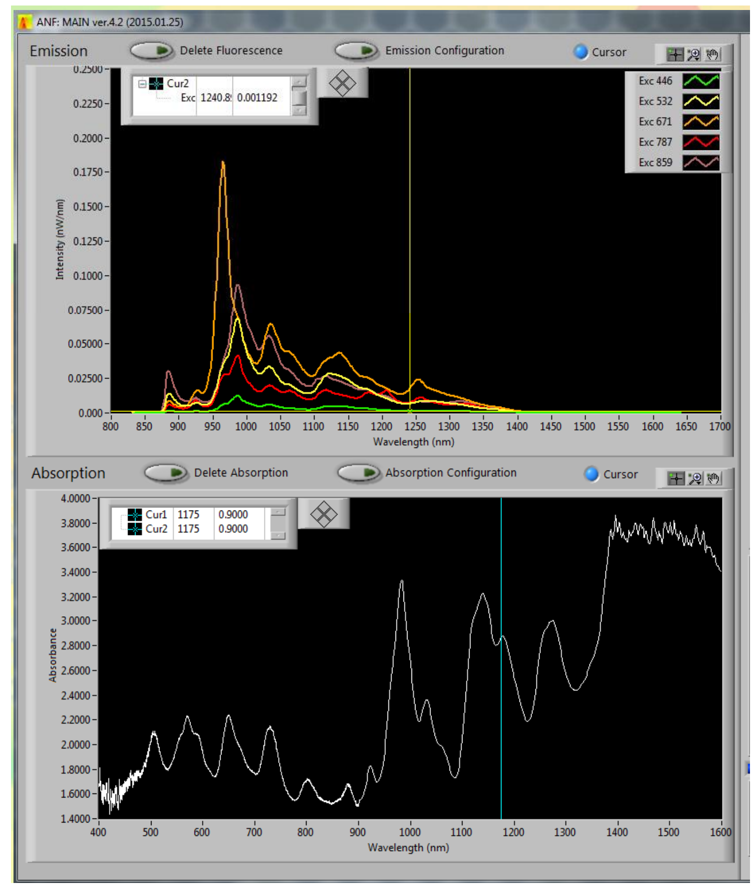
Visible (450-900 nm)
Near-Infrared (900-1600 nm)
Extended Near-Infrared (1600-1900 nm)
with up to 5 lasers for excitation

Absorption

Ultraviolet (210-400 nm)
Visible (400-900 nm)
Near-Infrared (900-1600 nm)
Extended Near-Infrared (1600-1900 nm)

Raman:

532 nm excitation (150-3000 cm^{-1})
671 nm excitation (150-3000 cm^{-1})
optimized for carbon nanomaterials



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NanoSpectralyzers: Customization Options

	NS1	NS2	NS3	Most Popular NS3
NIR emission and absorption	✓	✓	✓	✓
Visible absorption	✓	✓	✓	✓
Raman (1 excitation wavelength)		✓	○	✓
Raman (2 excitation wavelengths)			○	✓
Visible emission			○	✓
Extended NIR emission/absorption			○	
UV absorption			○	
External signal input port			○	
Laser output port			○	
Vertical sample scanning	○	○	○	
Reduced sample volume	○	○	○	

○ = Optional

Our most popular NS3 configuration includes all of the options of the NS1 and NS2 plus a few more!

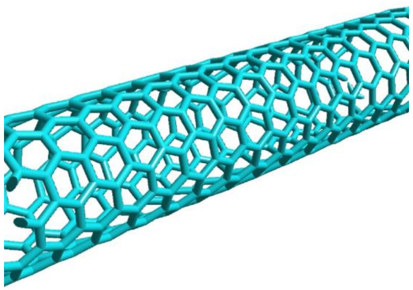
Since the spectroscopy modules, software interface, and nanotube fitting routines for the NS1 & NS2 are included in this NS3

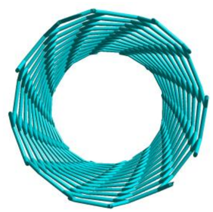
→ **understanding** the NS3 prepares you to use all 3 instrument models!

What types of nanomaterials?

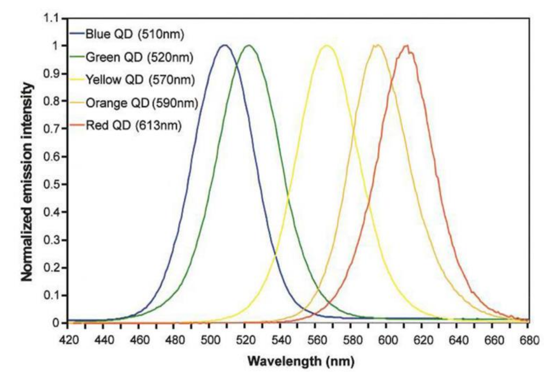
Metallic nanoparticles:
gold, silver, spheres, or rods

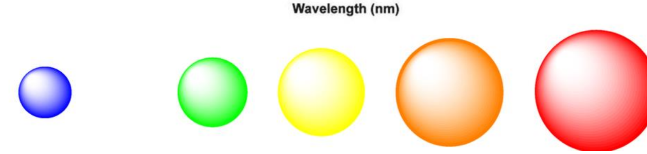
Carbon nanoparticles:
carbon nanotubes, graphene,



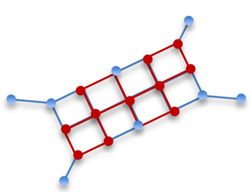


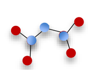
Quantum dots:
CdSe, PbS, etc



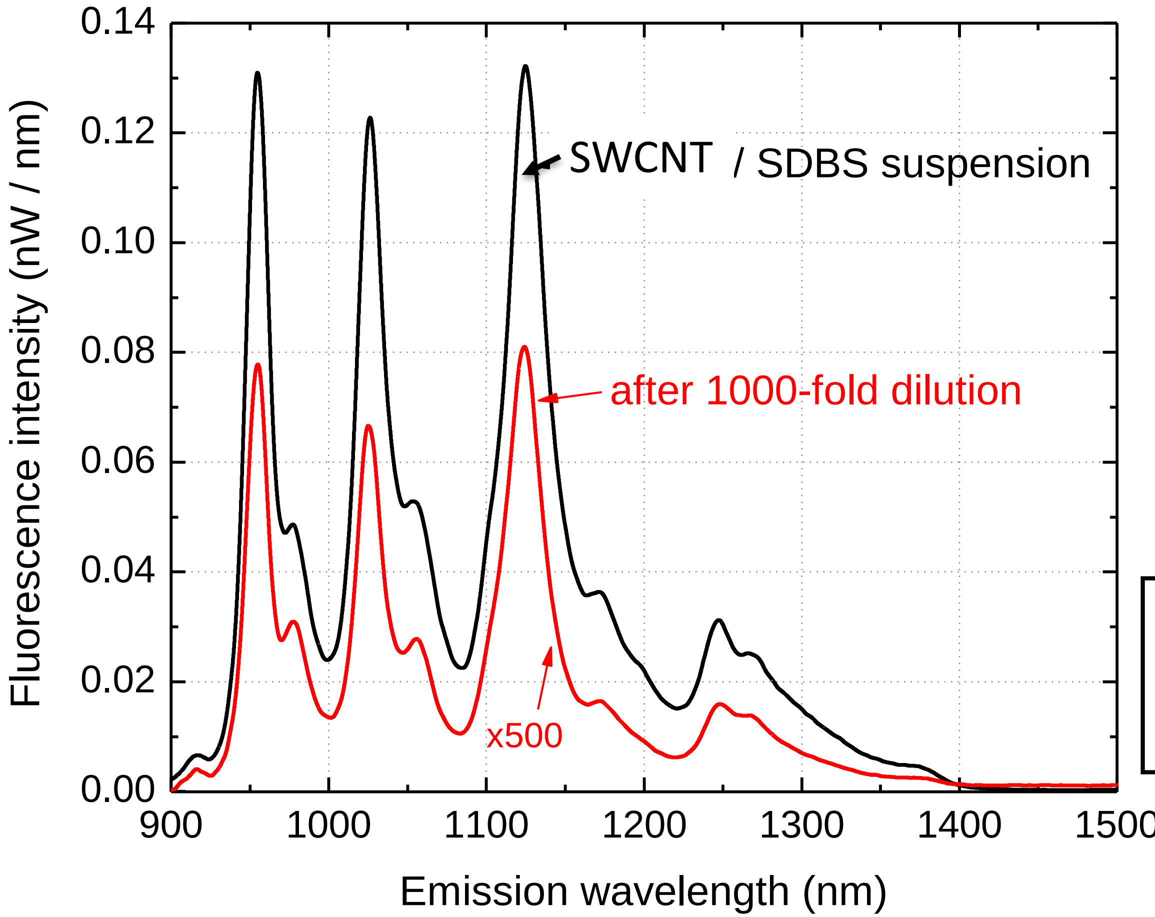


Other nanoparticles & general
spectroscopy applications
NaNdF4, NaHoF4, CuO, small molecules





The NS3 - High Sensitivity

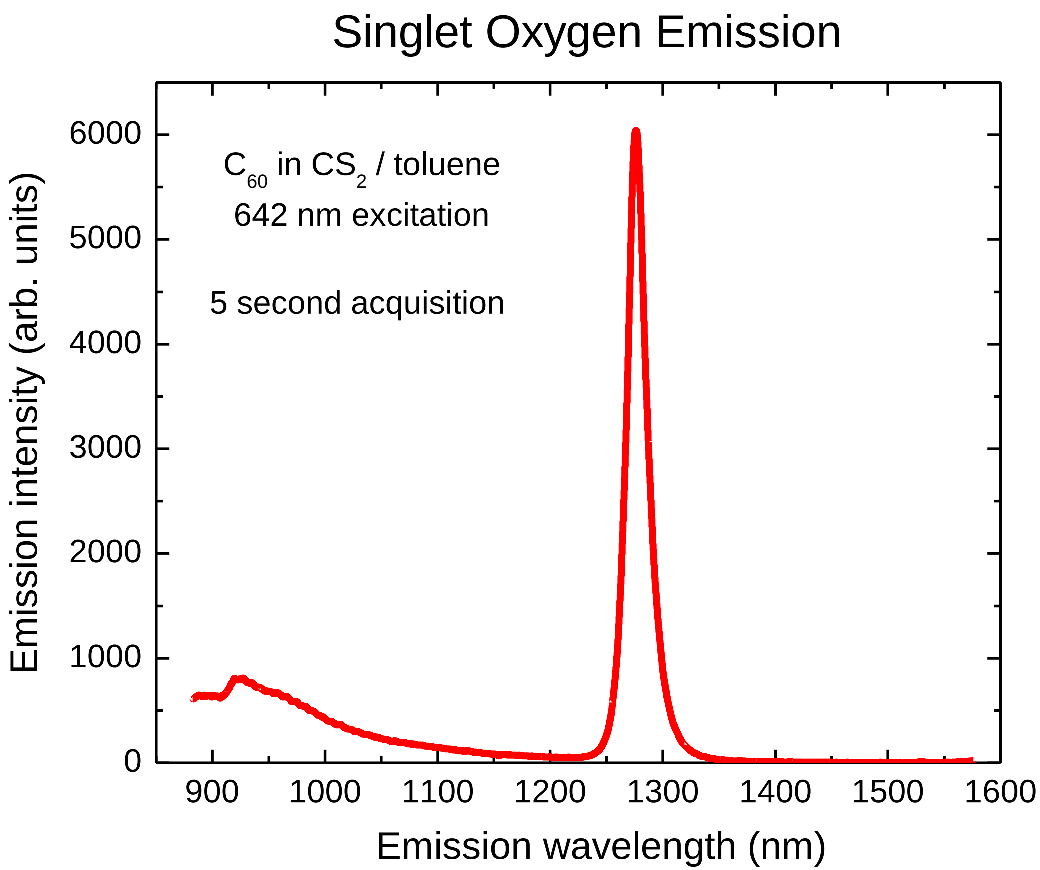


The NS3 NanoSpectralyzer is highly sensitive and can measure very dilute samples

Sensitive down to 10 pg of single-walled carbon nanotubes (10^{-10} by mass)

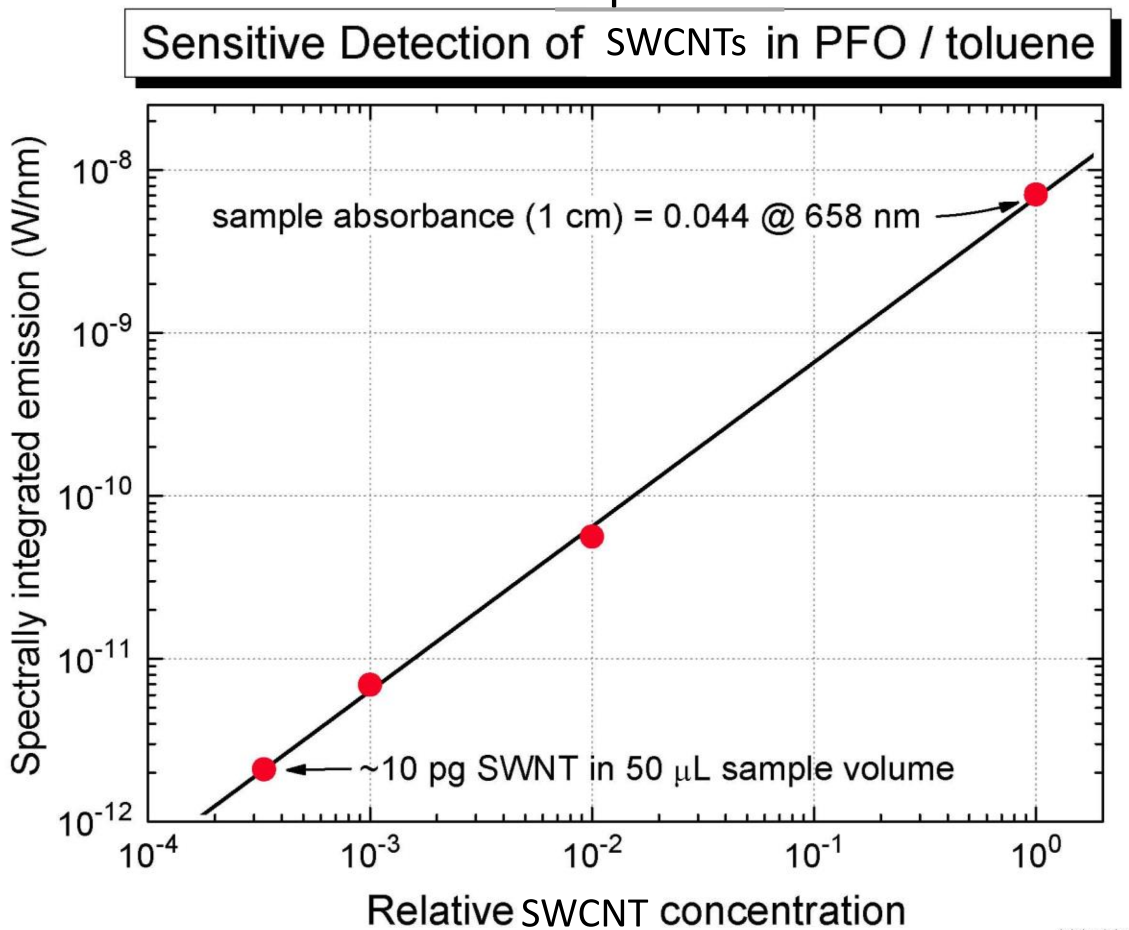
The NS3 - High Sensitivity

The NS3 is sensitive enough to measure phosphorescence from $^1O_2$

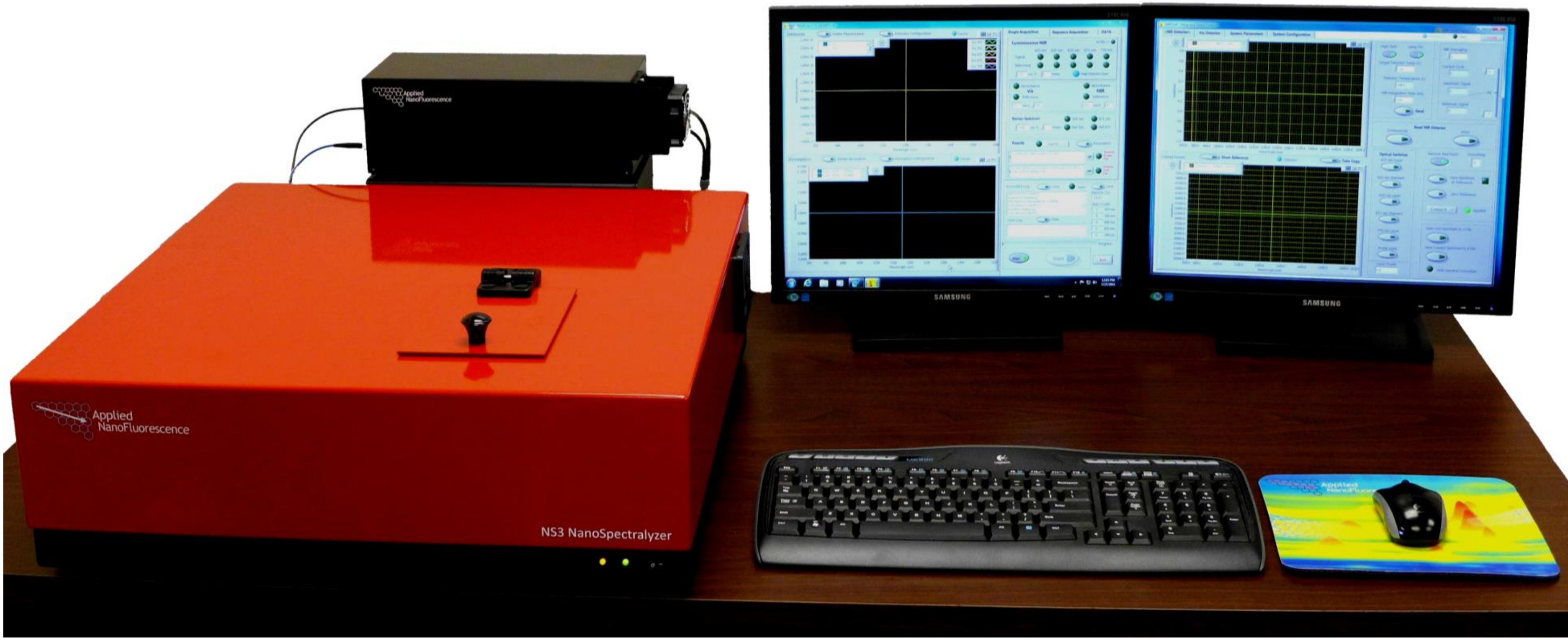


The NS3 - Large Dynamic Range

We have measured 5 orders of magnitude dynamic range for fluorescence samples



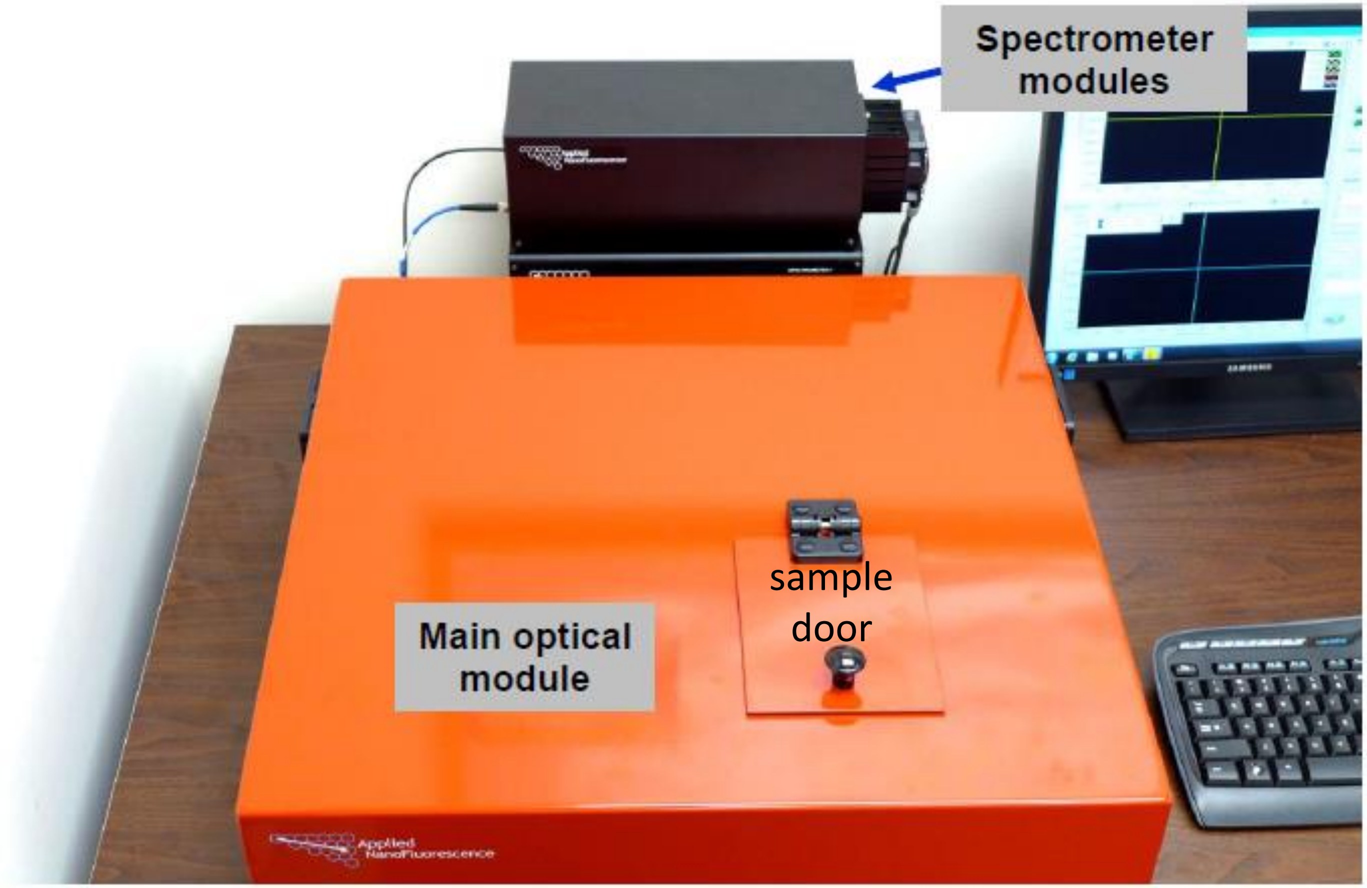
Model NS3 NanoSpectralyzer System Overview



The main system hardware includes the main optical module, external spectroscopy modules, and a control computer with preloaded software and dual monitors.

Peripherals included are a wireless keyboard and mouse, cuvettes, fluorescence standard, and an adapter set for smaller volume samples (≥120 µL).

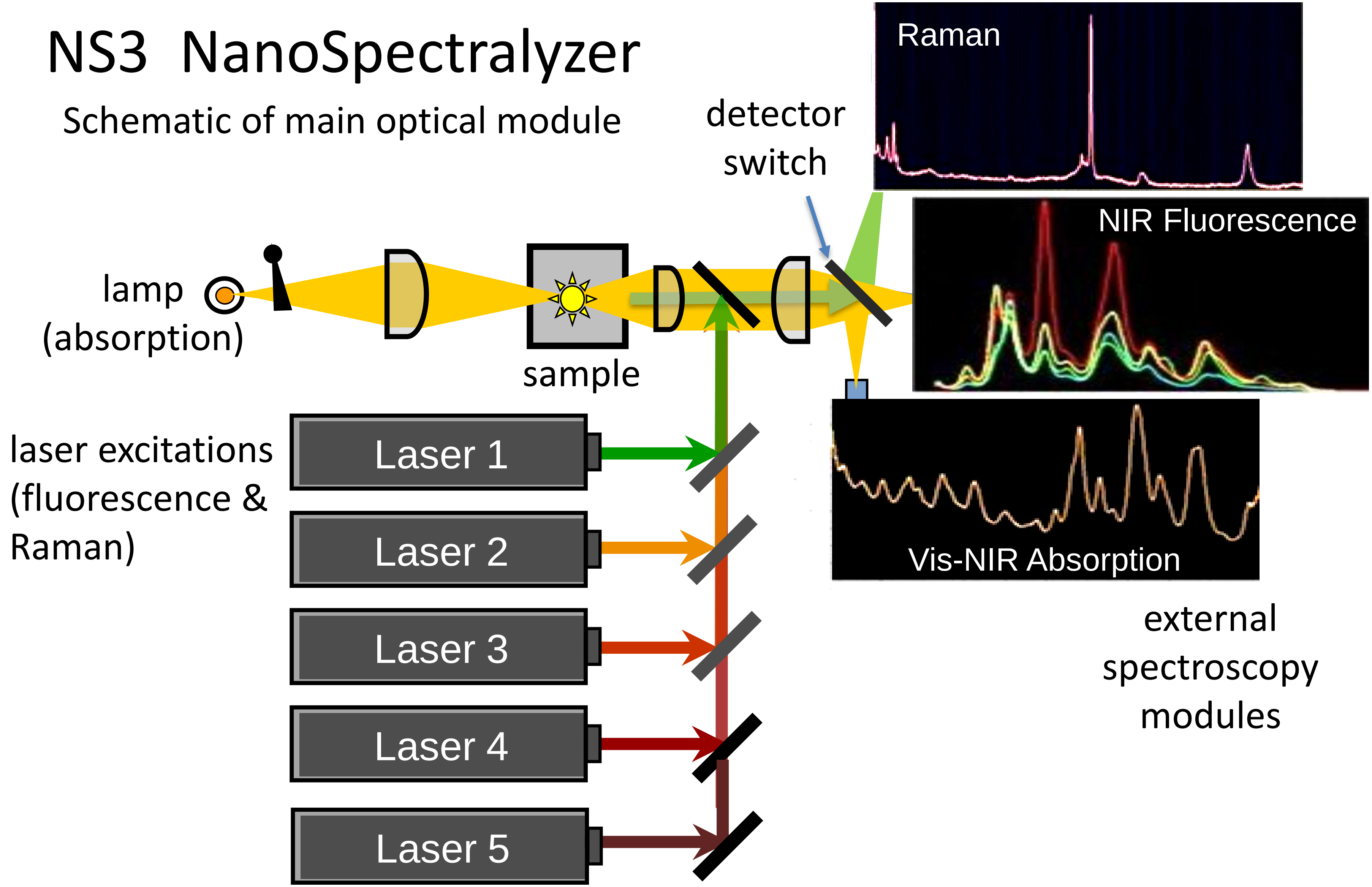
Model NS3 NanoSpectralyzer System



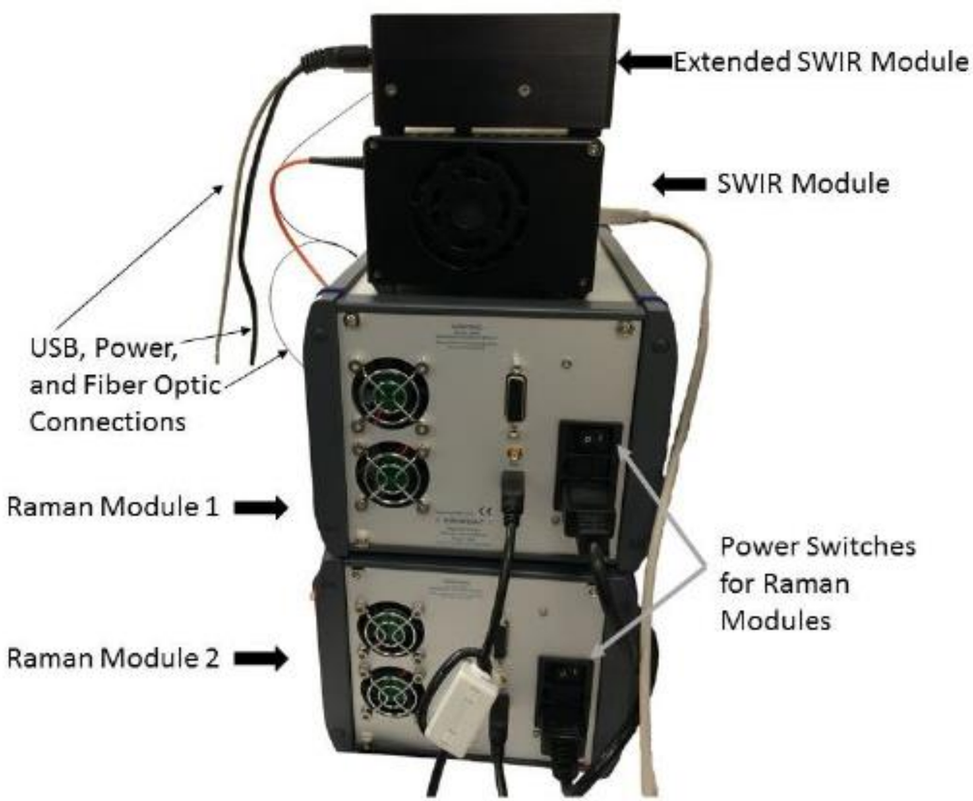
The main optical module contains all the excitation sources, electronics, and optical components to route all the spectroscopy acquisitions through a single cuvette. The cuvette is placed within the sample door shown above.

NS3 NanoSpectralyzer

Schematic of main optical module



NS3 Spectroscopy Modules – a closer look



Actual spectroscopy modules included with the system will depend on the specific configuration ordered

The external spectroscopy modules shown on the left receive light through fiber optic connections from the main optical module

The visible spectrometer module is located inside the main optical module

SWIR = short wavelength infrared
NIR = near-infrared

} our 900-1600 nm detectors are in the region where the near and short wavelength infrared overlap and the terms are used interchangeably

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NanoSpectralyze Software

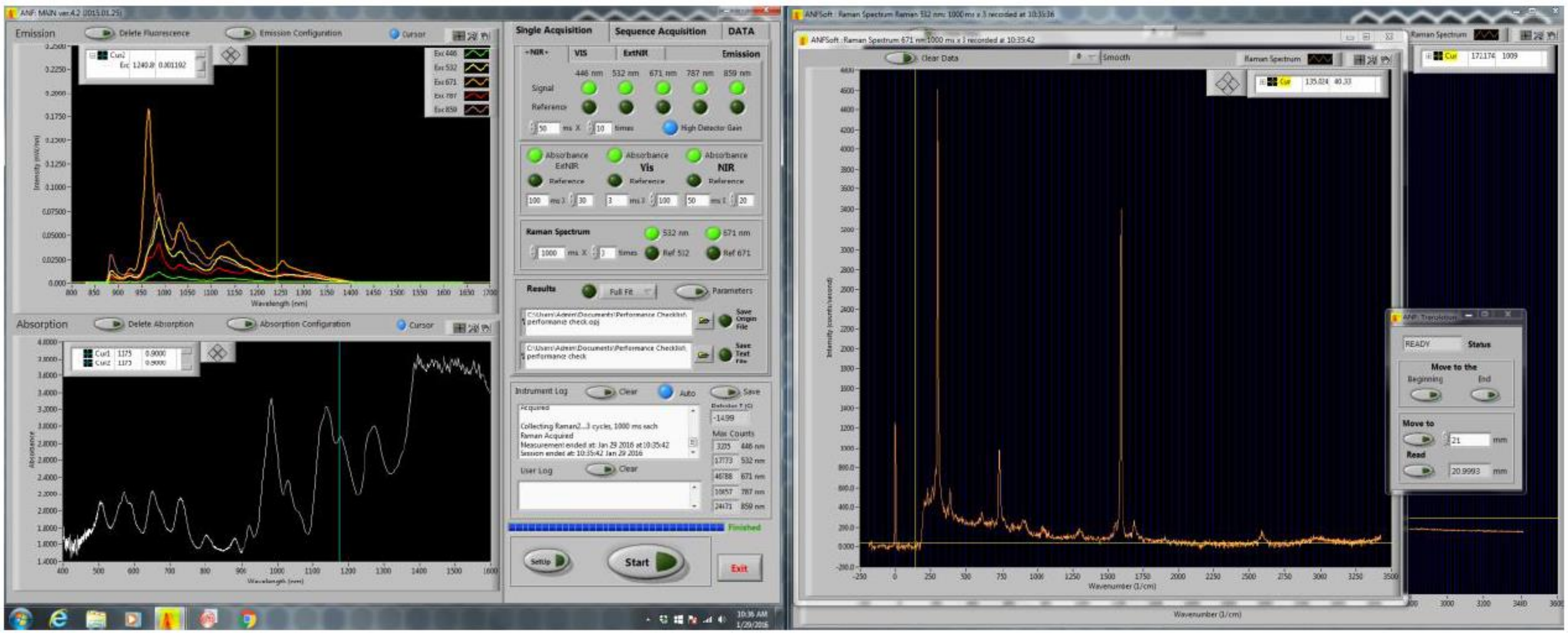
The ANFSoft Icon on the computer desktop will open the fully integrated NanoSpectralyze software that is used for system control, spectral acquisition, and data analysis

NanoSpectralyze software contains patented fitting routines to rapidly analyze the (*n*,*m*) and diameter distribution of semiconducting single-walled carbon nanotubes using their NIR fluorescence. Integrated into that fitting routine is the powerful scientific graphing and analysis software, Origin®.

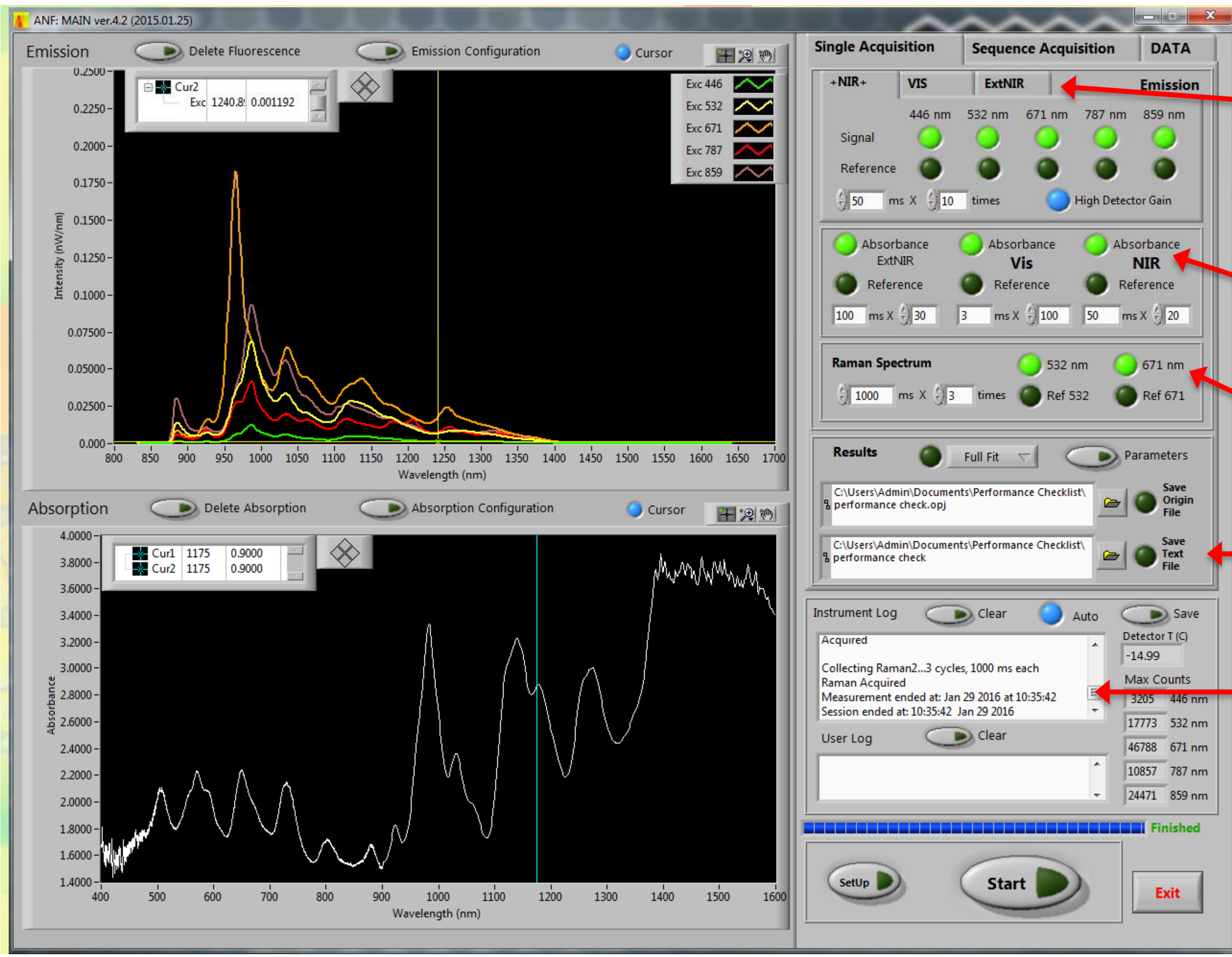
The NS1, NS2, and NS3 all include a licensed copy of Origin® and all the functionality that comes with it.
For more information see: www.originlab.com

NanoSpectralyze Software User Interface

Double clicking the icon will start system initialization, after a routine to check hardware status, the main control will appear.



Control Interface: Overview



Main control screen

Separate control tab for each fluorescence module

Absorption modules control

Raman modules control

File saving options

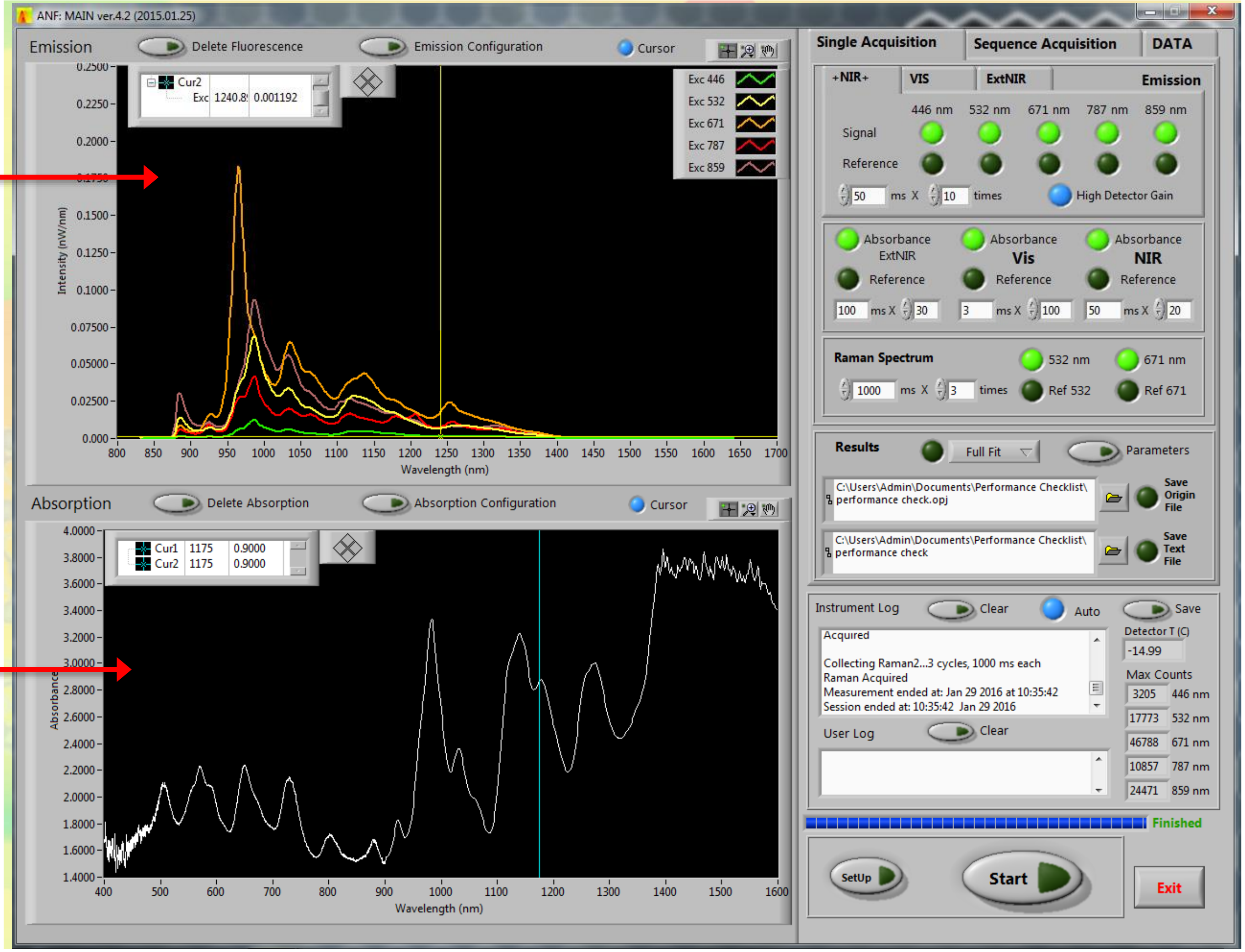
Instrument log with all acquisition parameters

Control Interface: Overview

Main control screen

Graphical output of NIR fluorescence data

Graphical output of NIR and visible absorption data



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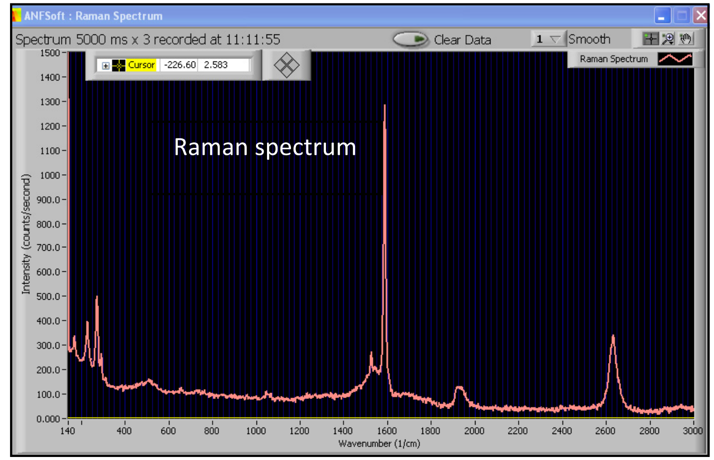
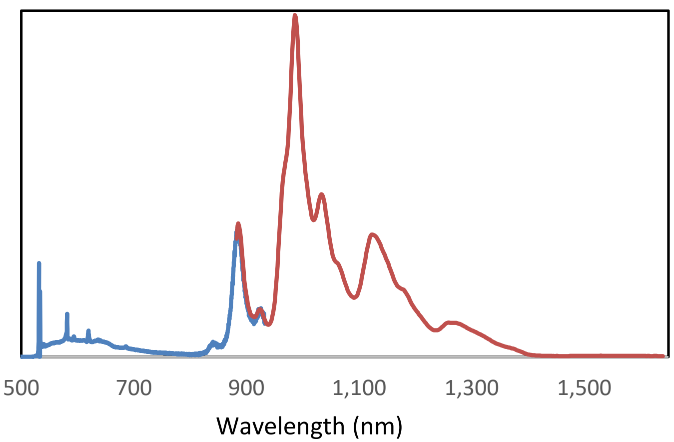
Control Interface: Raman and Visible Fluorescence

Raman and Visible fluorescence results display in separate floating windows

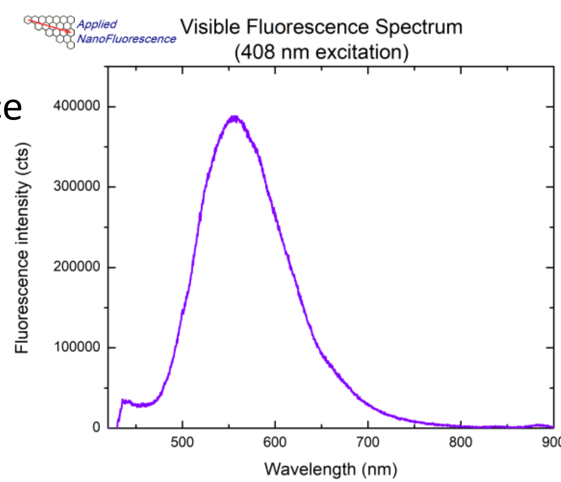
Floating window displaying graphical output of Raman data



Graphical output of visible emission data (blue) overlaid with NIR emission spectra (red) with 532 nm excitation of single-walled carbon nanotubes



Visible fluorescence spectrum of fluorescein in antifreeze with 408 nm excitation



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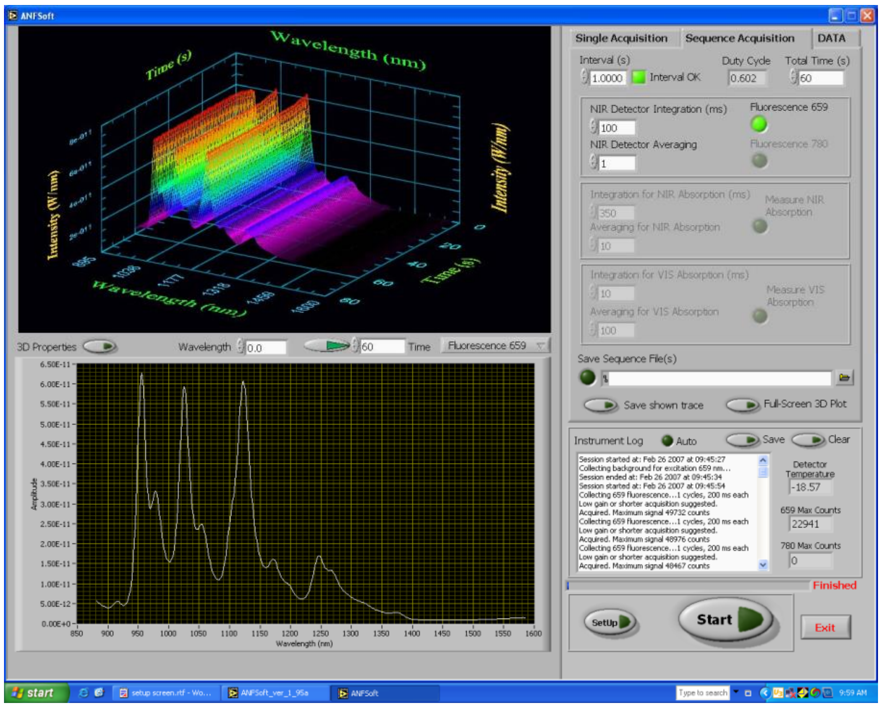
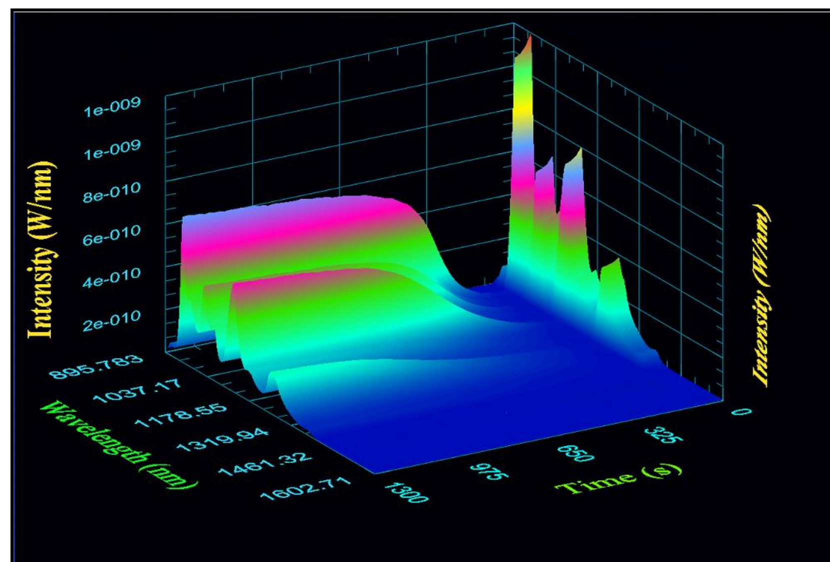
Control Interface: Sequence Acquisition



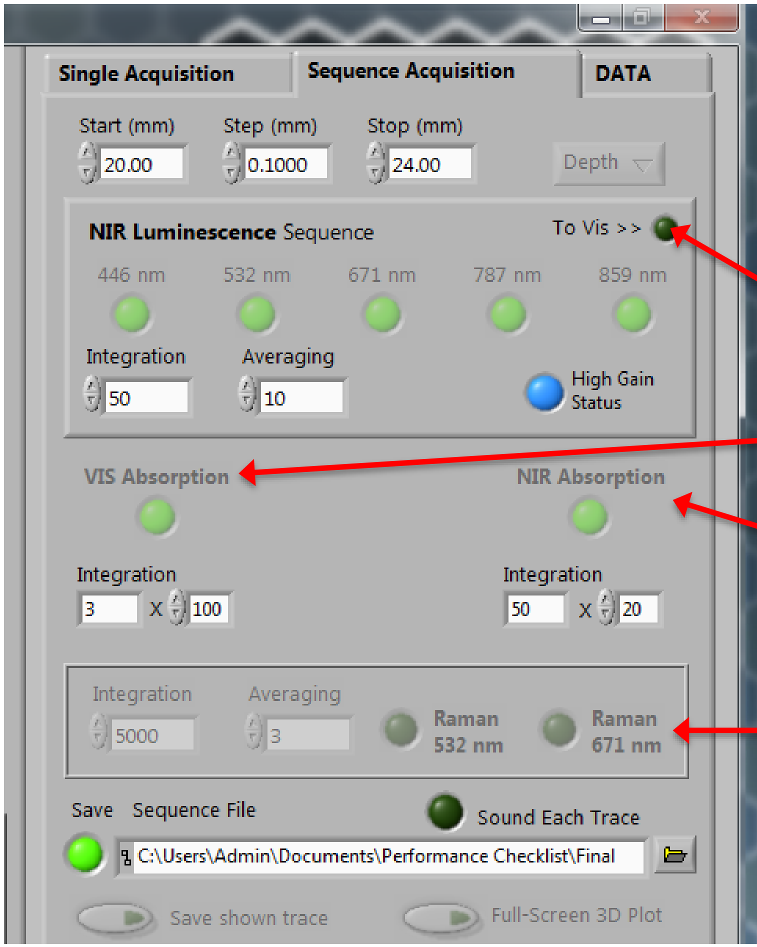
Sequence acquisition tab located on main control screen

Versatile time settings allow measurements of rapid or slow kinetic processes (from 10 spectra per second to obtaining a single spectra every 15 minutes over a day or longer)



Powerful 3D interactive graphical output of sequence data that you can rotate and reposition using the mouse

Control Interface: Sequence Acquisition



Sequence acquisition is available for any spectroscopy module included in the system not just NIR Fluorescence:

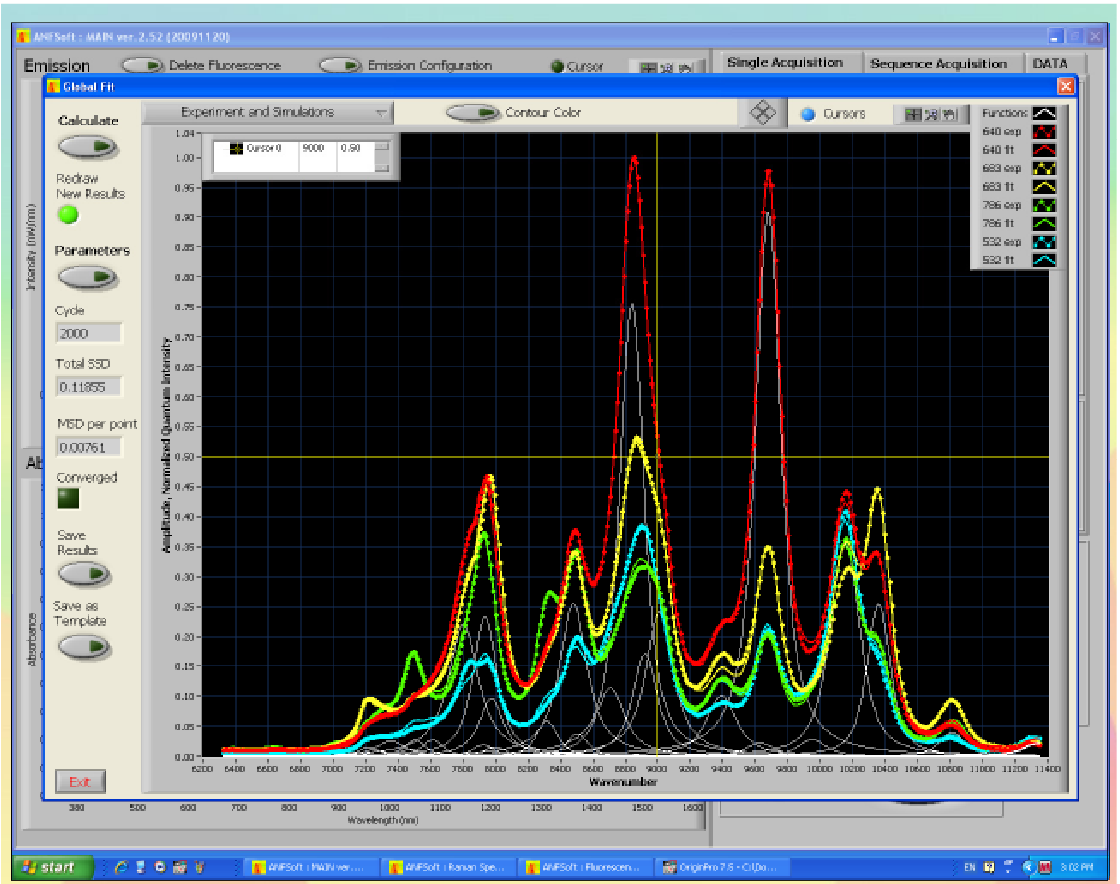
Visible Fluorescence

Visible Absorption

NIR Absorption

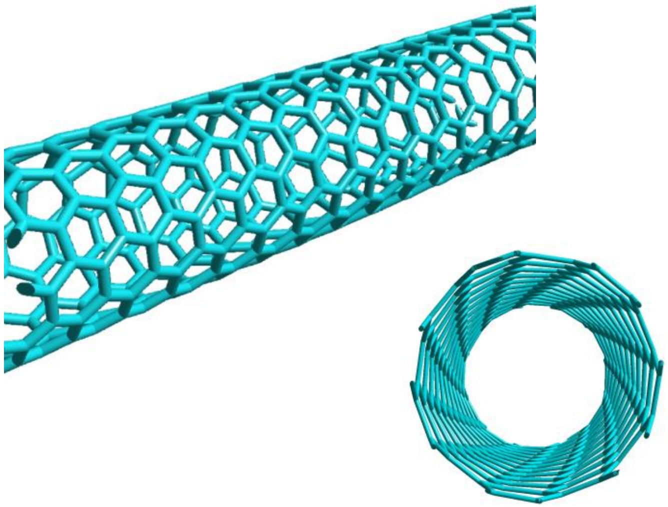
Raman

Specialty Analysis Software for Single-Walled Carbon Nanotubes



NIR emission data is spectrally fit dozens of semiconducting (*n*,*m*) species using the very latest in scientific research results

What are Single-Walled Carbon Nanotubes (SWCNTS)?



Properties:

Material: Carbon

Typical diameter: 1 nm

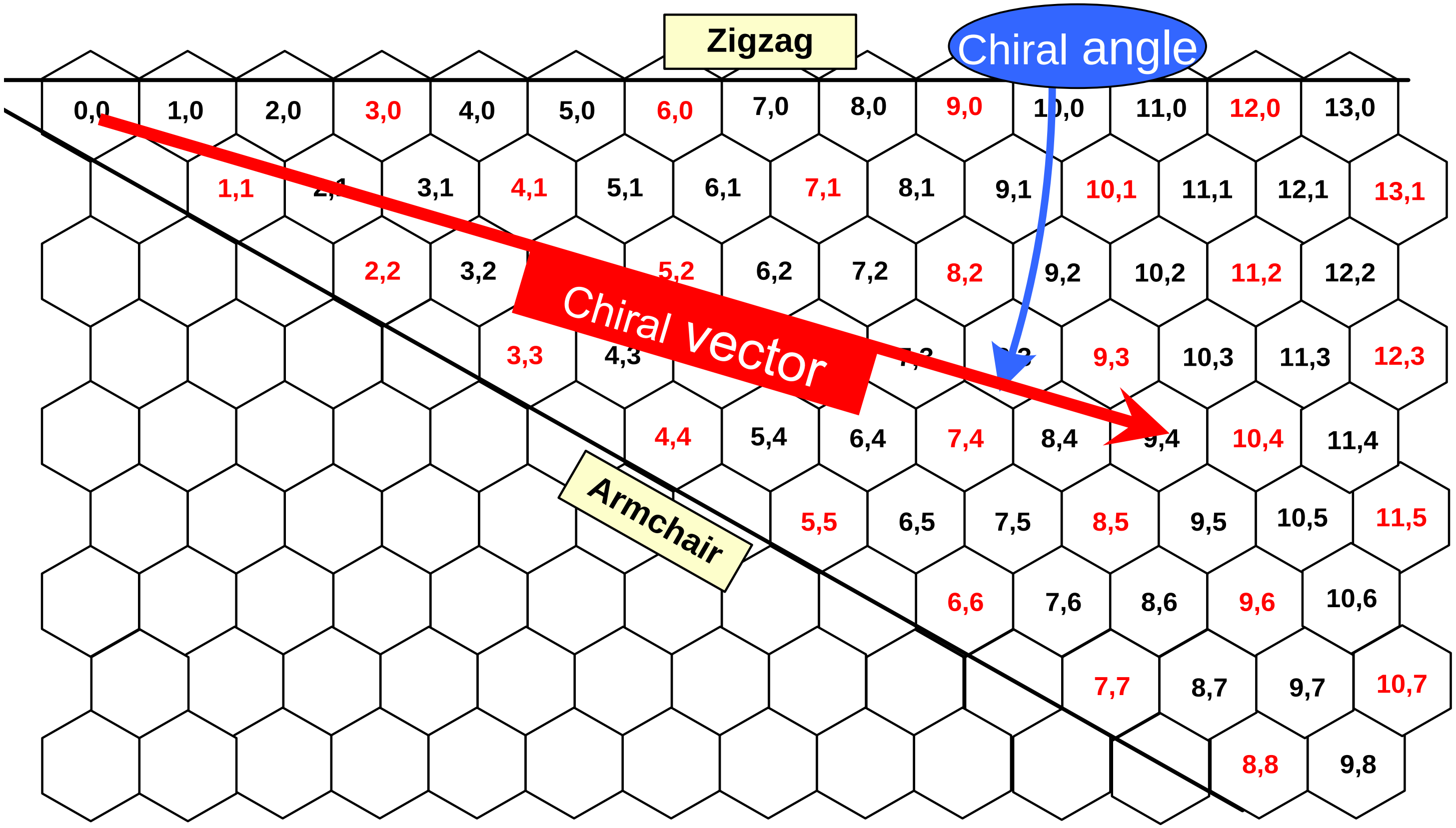
Typical lengths: 100 – 1000's nm (large aspect ratios)

Electrical transport: metallic or semiconducting

Optical transitions: p-p bands,*
direct band-gap semiconductors

Single-walled carbon nanotubes have a graphitic structure in the form of a cylinder a single atom thick. These carbon cylinders can be formed with a variety of diameters and **Multi-walled carbon nanotubes** are composed of multiple concentric single-walled carbon nanotubes.   Near-infrared fluorescence can be observed from a subset of single-walled and few-walled structures of carbon nanotubes.

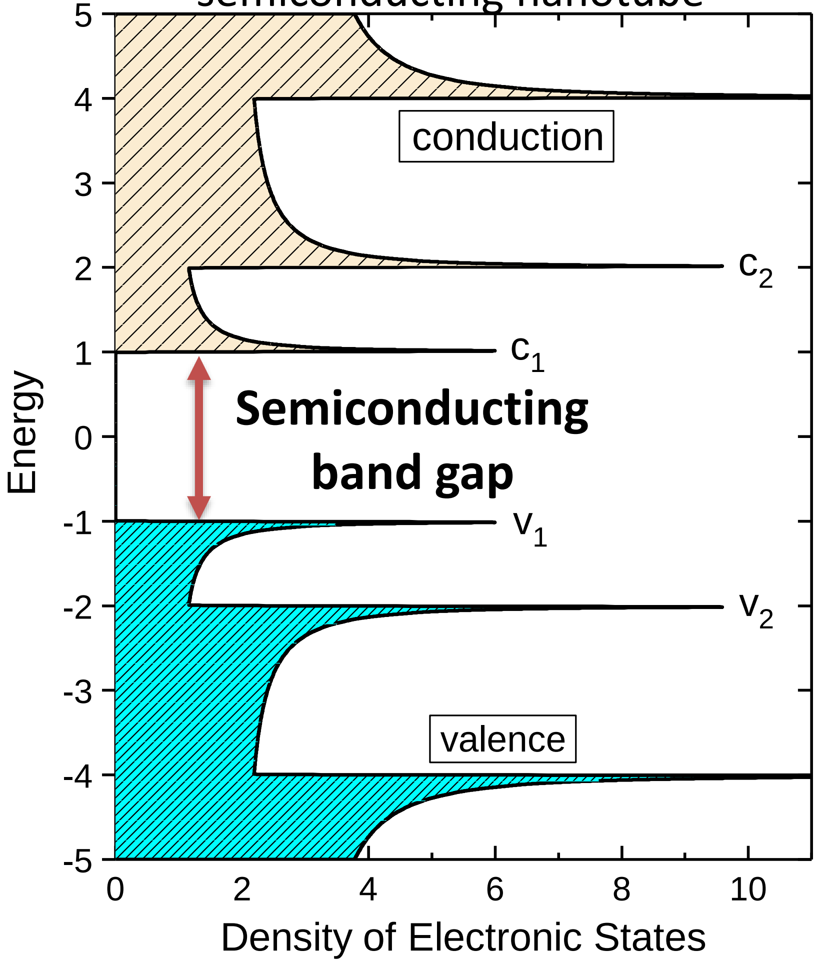
Each possible nanotube structure is labeled by two integers, (*n*,*m*), that uniquely define its diameter and chiral angle.



$$\textit{nanotube diameter} = (n^2 + m^2 + nm)^{1/2}\ 0.0783\ nm$$

Metallic vs. Semiconducting SWCNTS

Electronic band structure of a semiconducting nanotube



Metallic and Semiconducting nanotubes can be identified by their (n,m) designation.

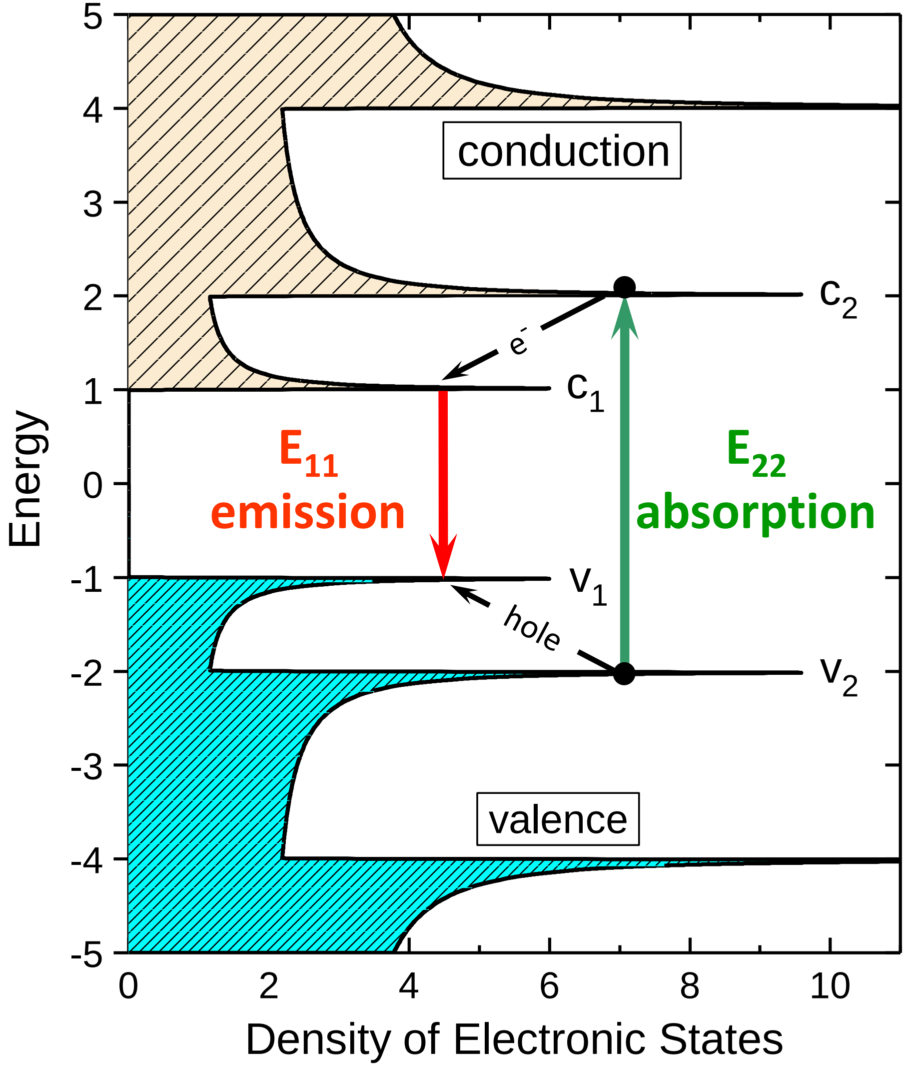
Metallic nanotubes: the value *n - m* is divisible by three

Semiconducting nanotubes: the value *n - m* is NOT evenly divisible by three.

Approximately 2/3rds of SWCNTs are semiconducting

The semiconducting nanotubes fluoresce in the near-infrared

Nanotube Fluorescence



Semiconducting and metallic nanotubes both **absorb light in the visible region** (E_{22} absorption). The wavelength of visible light absorbed varies with each nanotube structure.

Only semiconducting nanotubes will **fluoresce in the near-infrared region** (band-gap E_{11} emission). The wavelength of the near-infrared fluorescence emitted again varies with nanotube structure.

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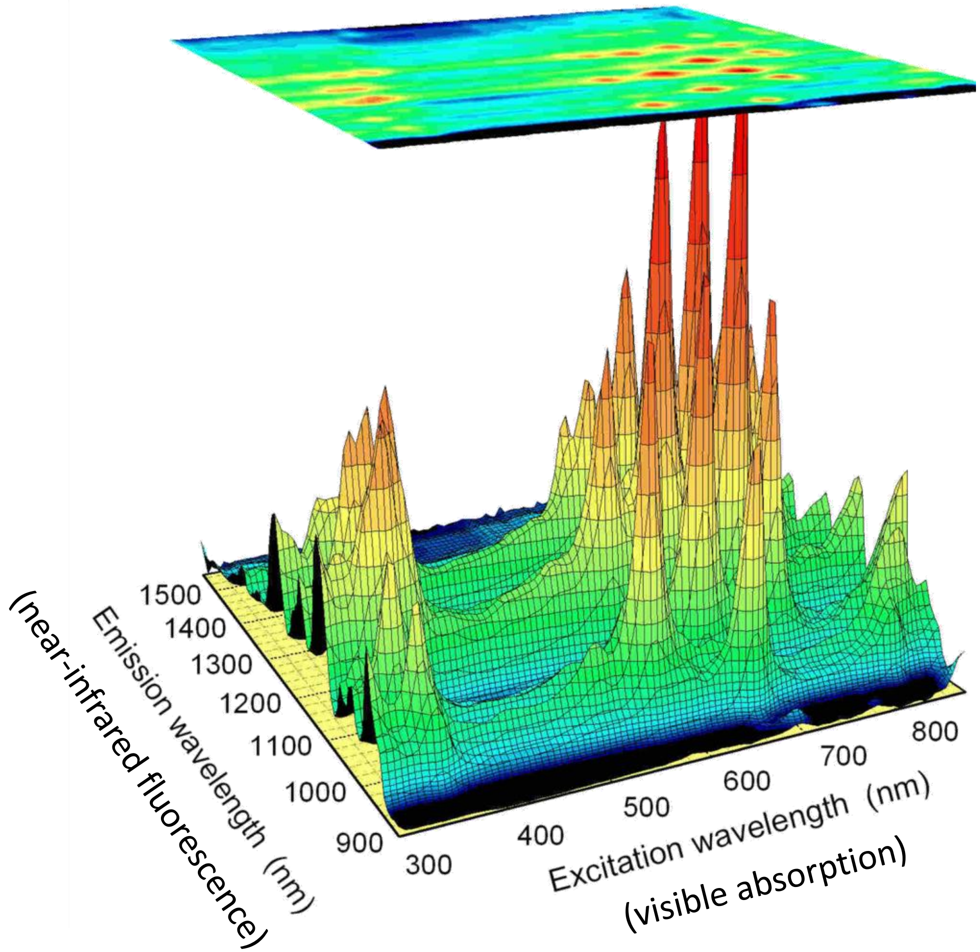

When we scan the visible excitation wavelength and capture the emission in the near-infrared we obtain a separate peak for each semiconducting (*n,m*) structure.

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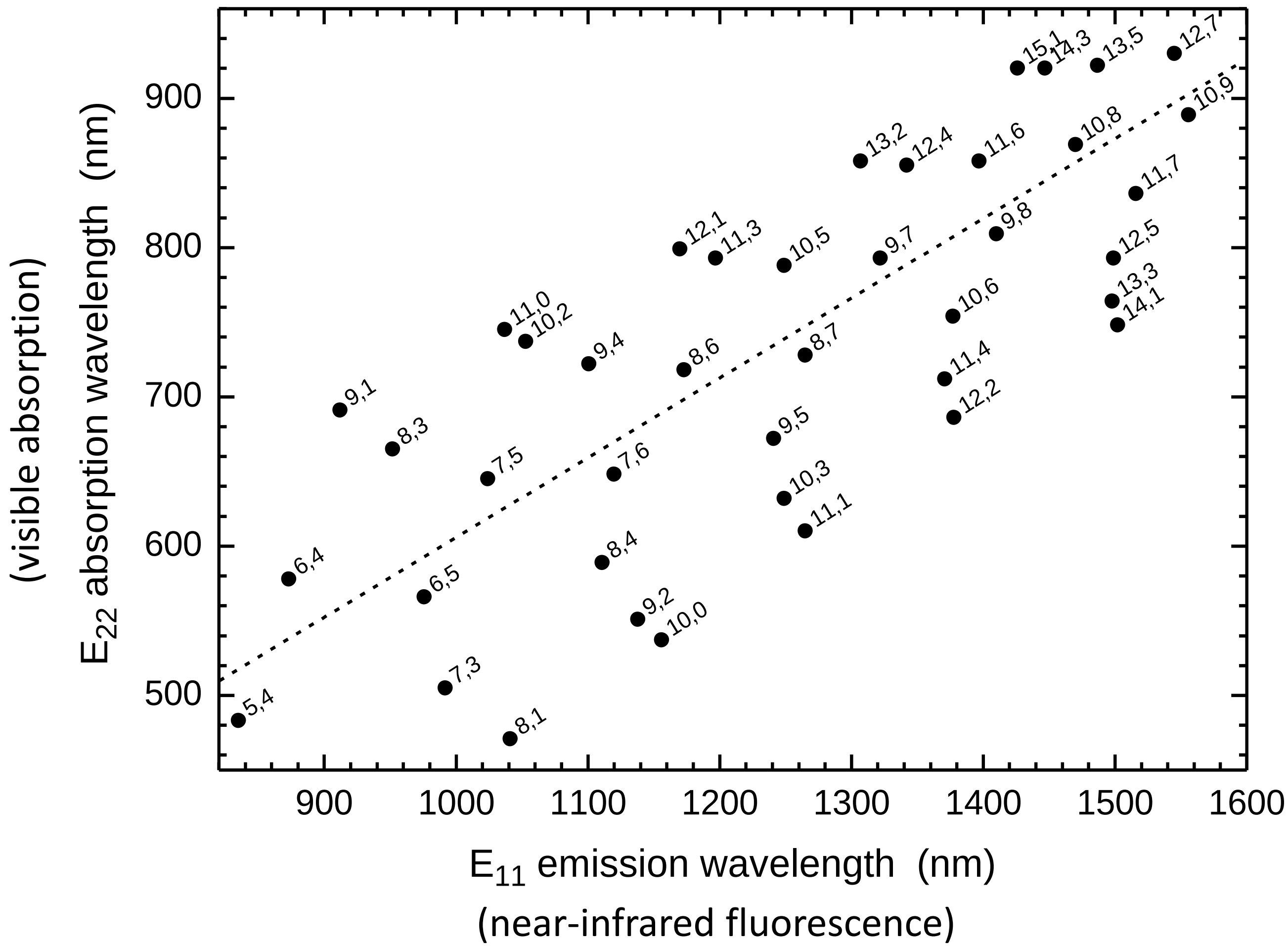
The (*n,m*) designation for the nanotube structure that produces each of these spectral peaks have been identified



Why do we need efficient (n,m) analysis?

- To refine and monitor SWCNT production processes
- To check quality and composition of SWCNT in industrial applications
- To check quality and composition of SWCNT in research applications
- To develop methods for sorting SWCNT mixtures by structure
- To aid research into structure-dependent processes

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Benefits of Fluorimetric Analysis

- High sensitivity
- Simple sample preparation
- Excellent (*n,m*) identification
- High selectivity against impurities, bundles, imperfect tubes
- No background subtraction needed in analysis (unlike absorption methods)
- Relatively simple instrumentation

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Typically a lamp is used to scan the visible absorption wavelengths

Disadvantages of using a lamp

- Slow data acquisition
- Limited sensitivity
- Tedious manual data reduction and interpretation

Is there a more efficient approach? Yes!

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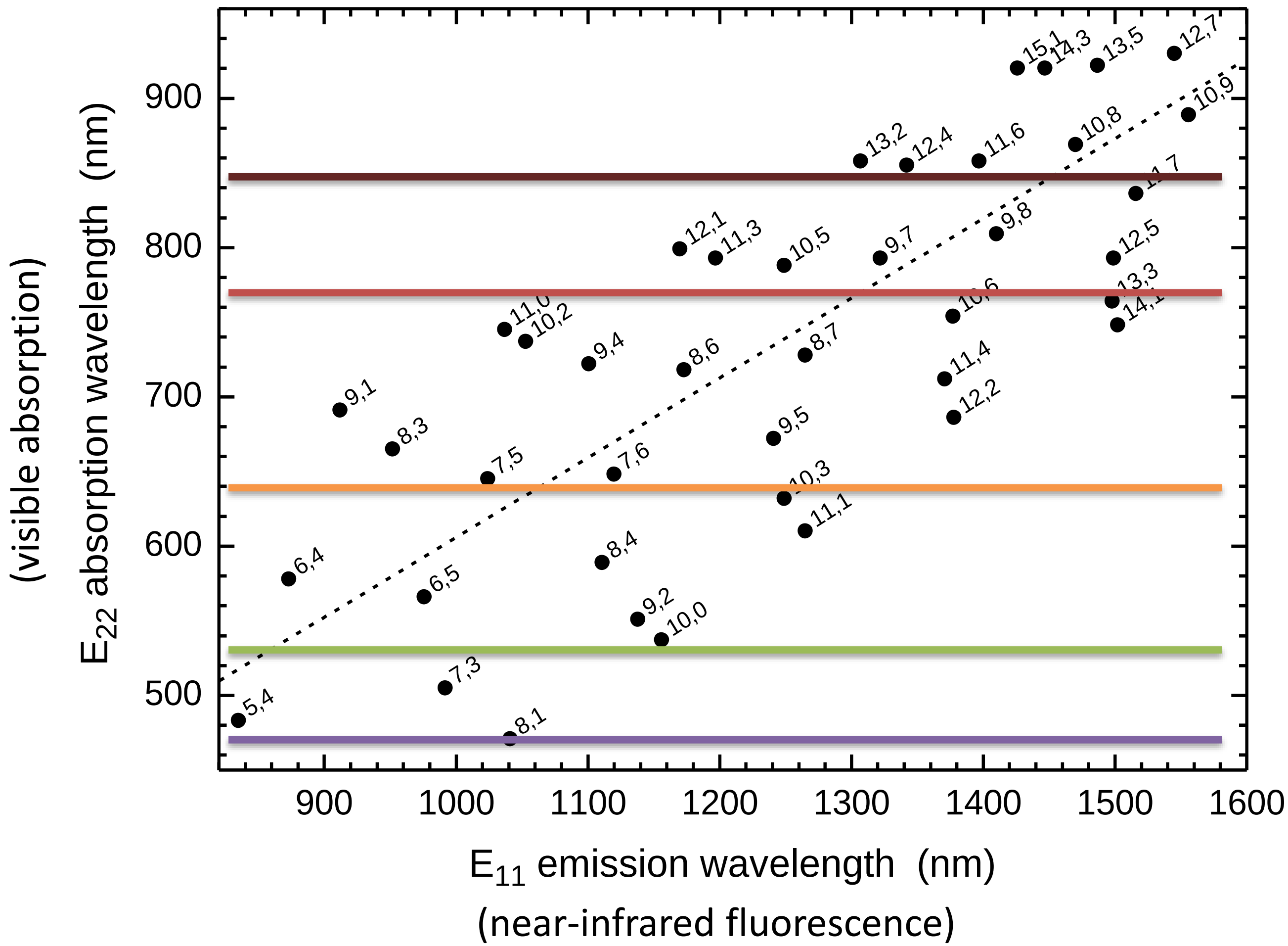
Instead of scanning the entire visible absorption region we use 5 distinct laser excitation wavelengths and interpolate the regions in between.



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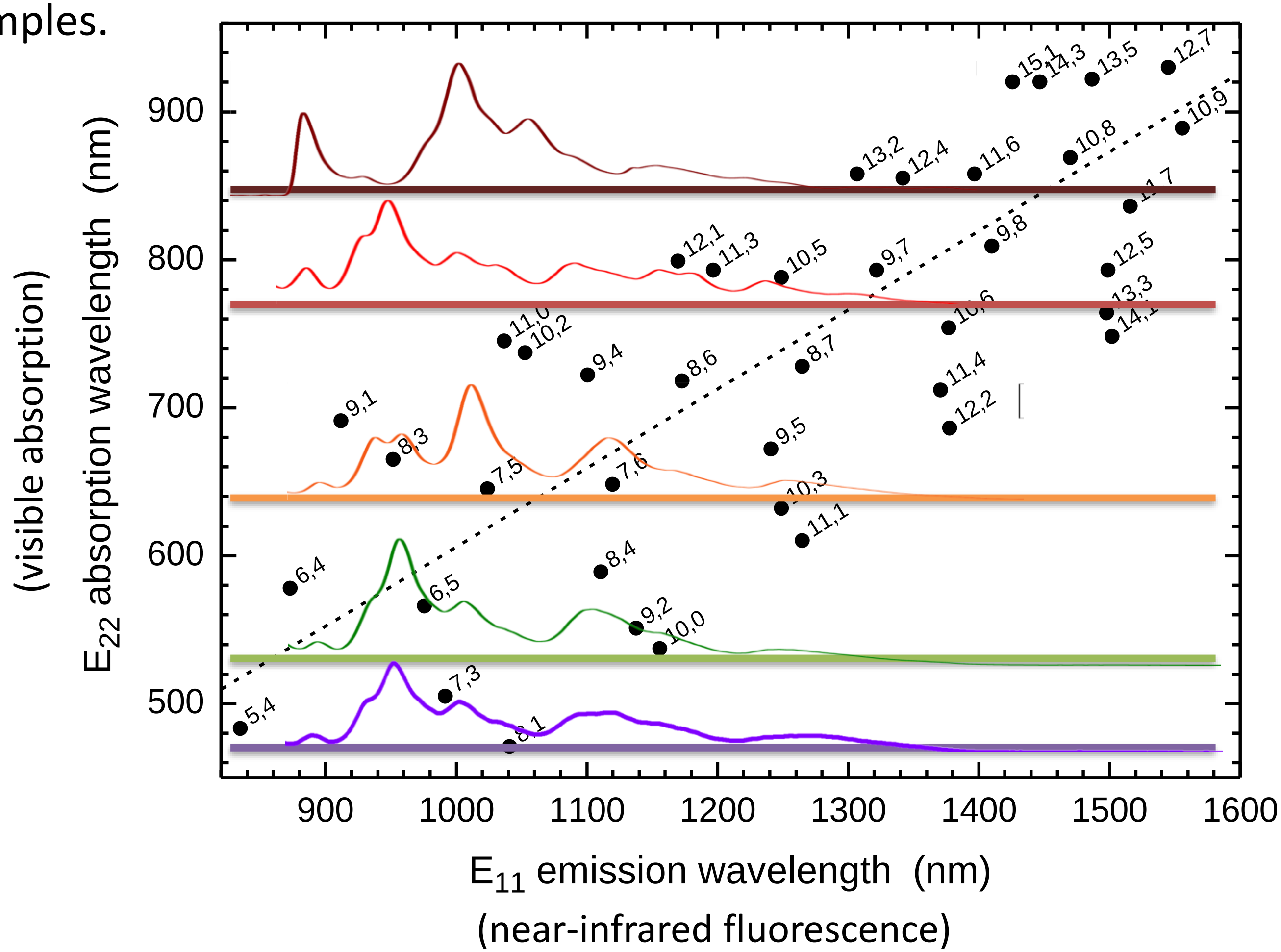
We can obtain these 5 fluorescence spectra in less than a second – a full scan of the visible absorption region can take 10's of minutes to hours! And because we use much more powerful laser excitations we can rapidly characterize very dilute samples.



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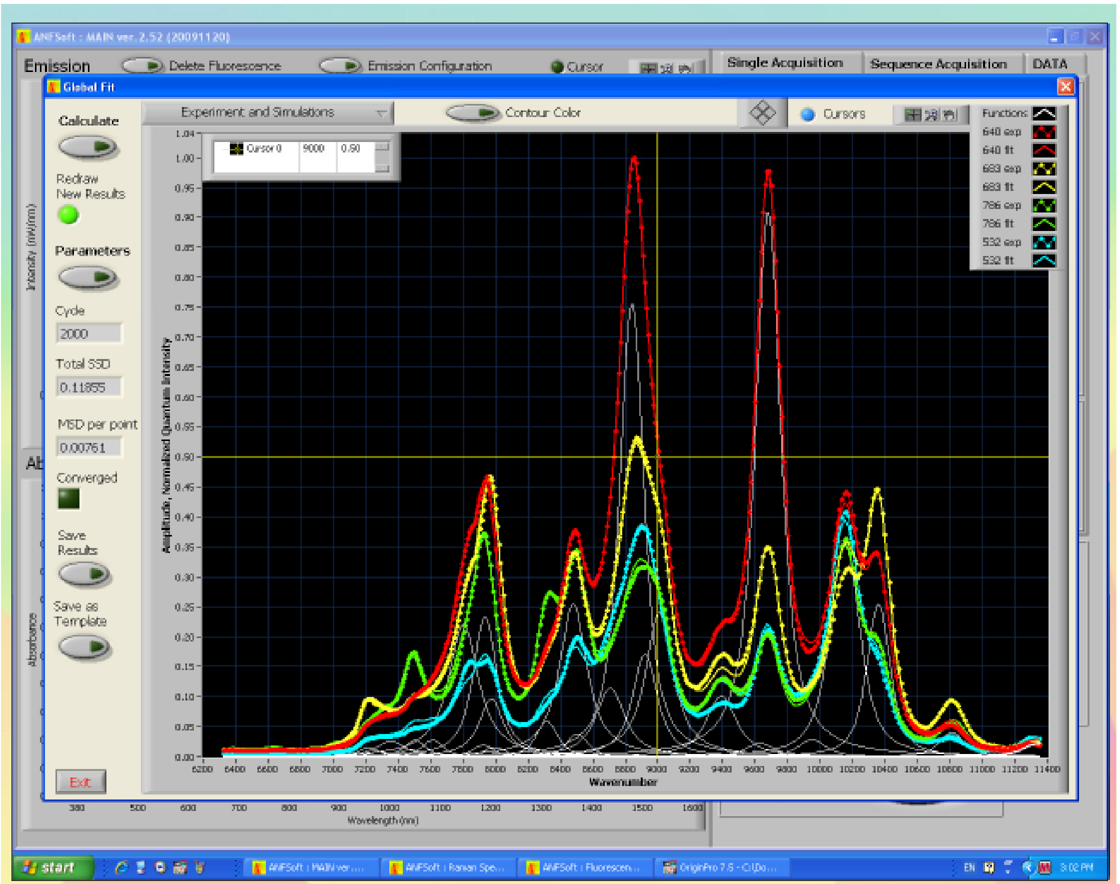
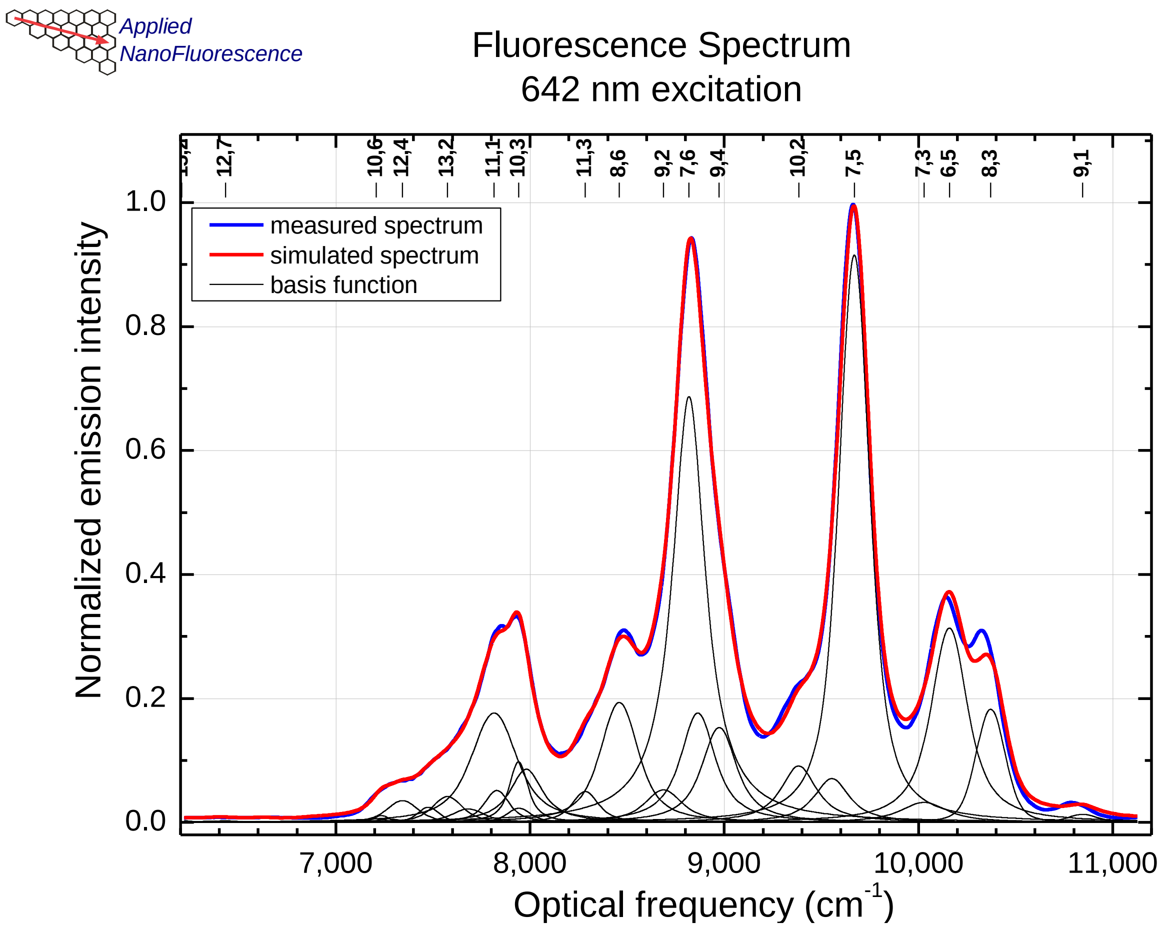
Rapid fitting deduces (*n,m*) composition from the combined fluorescence spectra



We then use the very latest scientific research of SWCNT optical properties to deduce the spectral intensities for all of the semiconducting (*n*,*m*) structures from these 5 fluorescence spectra.

We have designed fitting templates for a variety of nanotube samples and we work with individual customers to develop new templates to fit their applications.

Fitting process to deduce (*n,m*) distribution



There is a basis function (shown in black) for each (*n,m*) structure of carbon nanotube. The peak areas for each basis function is used to calculate the relative abundance of these structures.

The fitting is conducted on the fluorescence spectra for all 5 excitation wavelengths simultaneously to obtain consistent results

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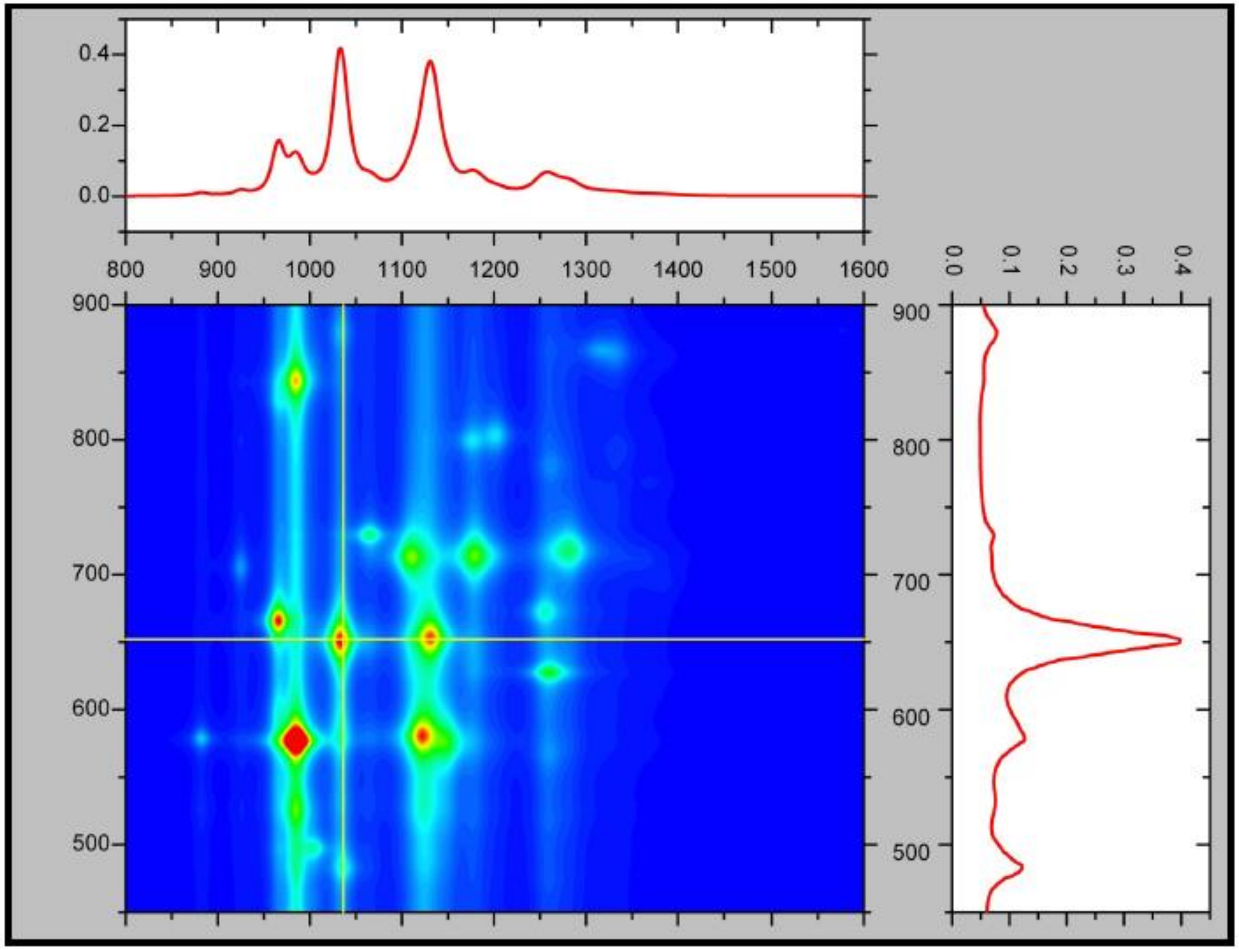
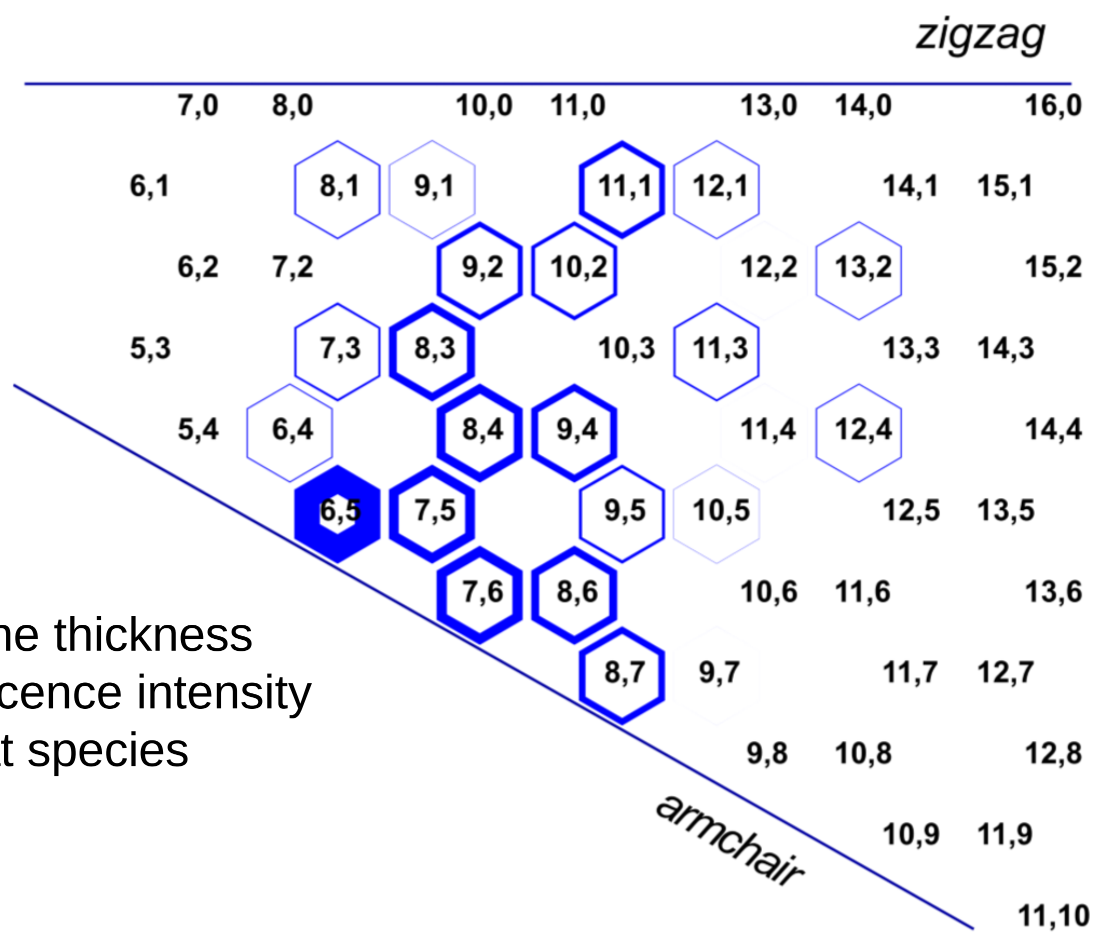
This calculated data is used to reconstruct the full visible scan of the nanotube sample from the 5 individual excitations

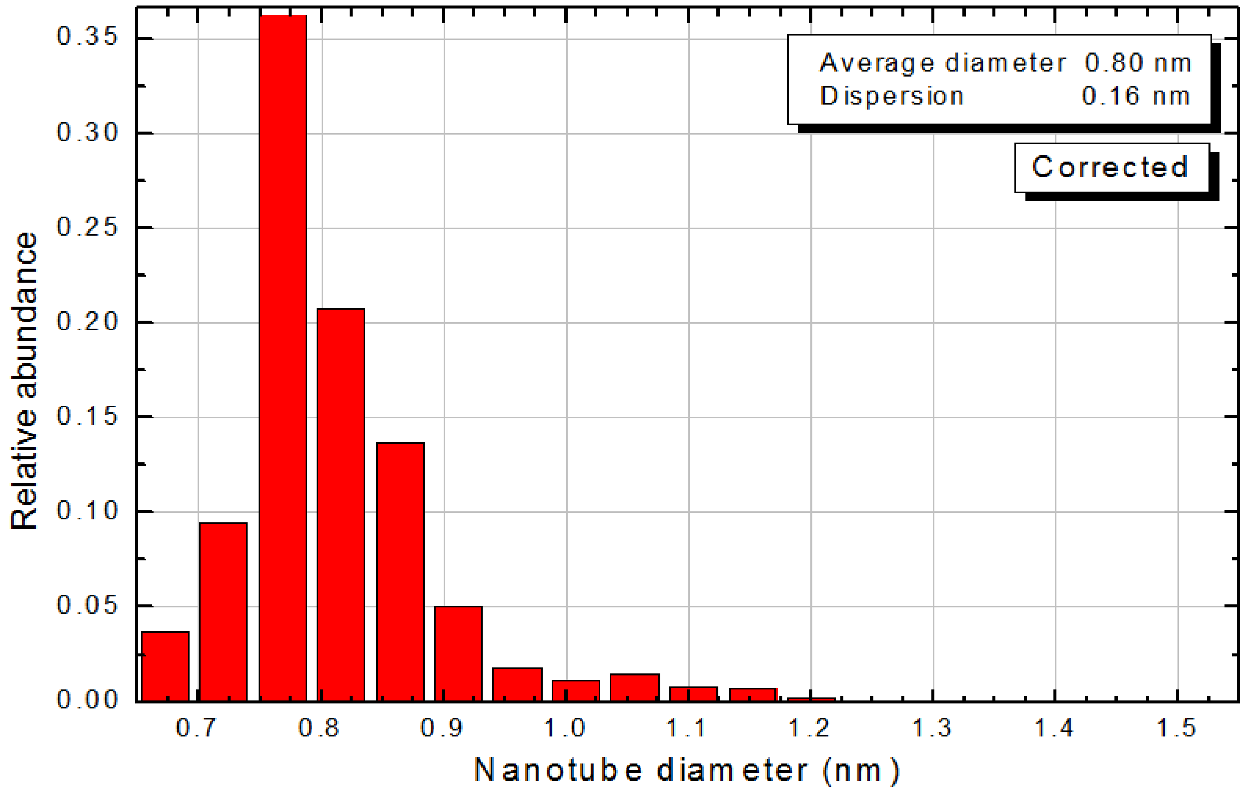


It is also used to create the graphene sheet map showing relative abundancies of the (*n,m*) structures



Hexagon line thickness shows fluorescence intensity from that species

And to calculate the nanotube diameter distribution in the sample



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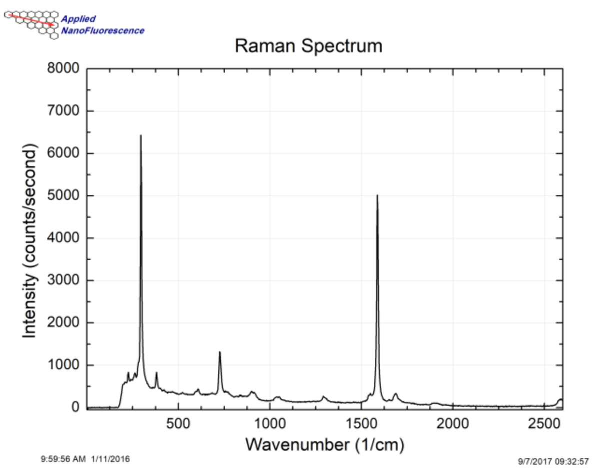
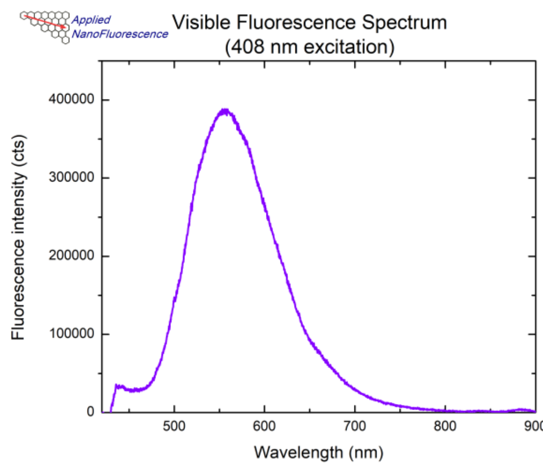
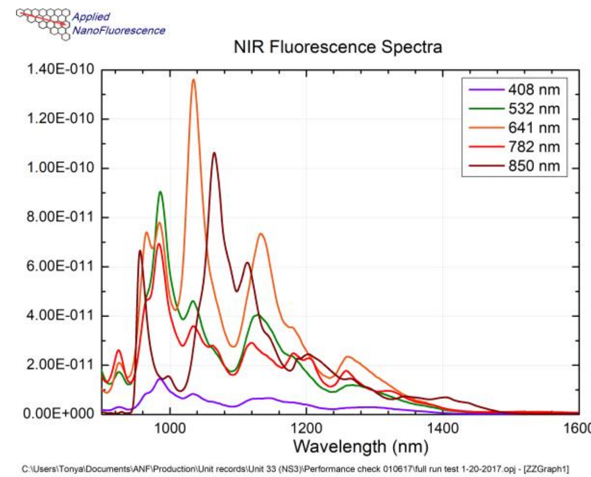
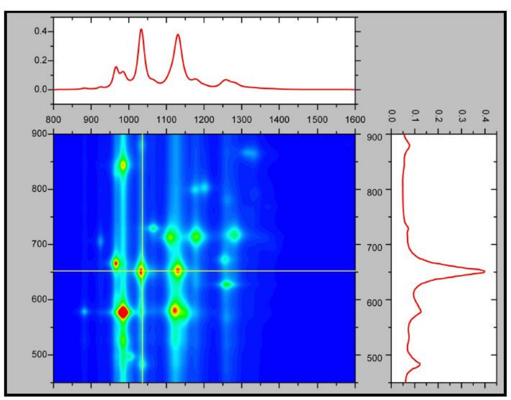
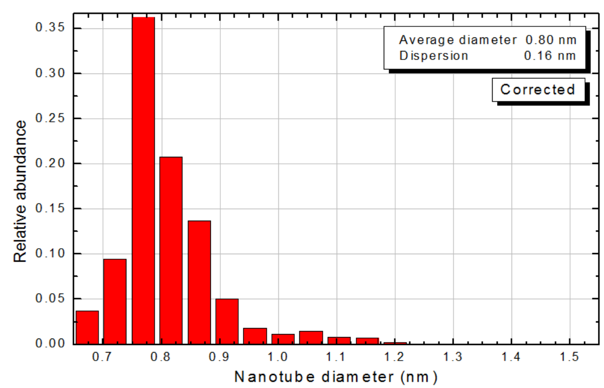
The spectral data and analysis for a sample are automatically compiled in an Origin file with publication ready plots

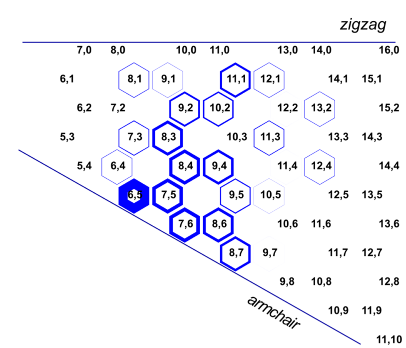












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Additional Options for NanoSpectralyzers:

- Reduced volume – sample volumes down to 50 µL
- External signal input – route external signals into the NanoSpectralyzer (depends on signal type and compatibility)
- Laser output port – fiber optic connection to use laser sources with external devices
- Vertical translation – details on next slide

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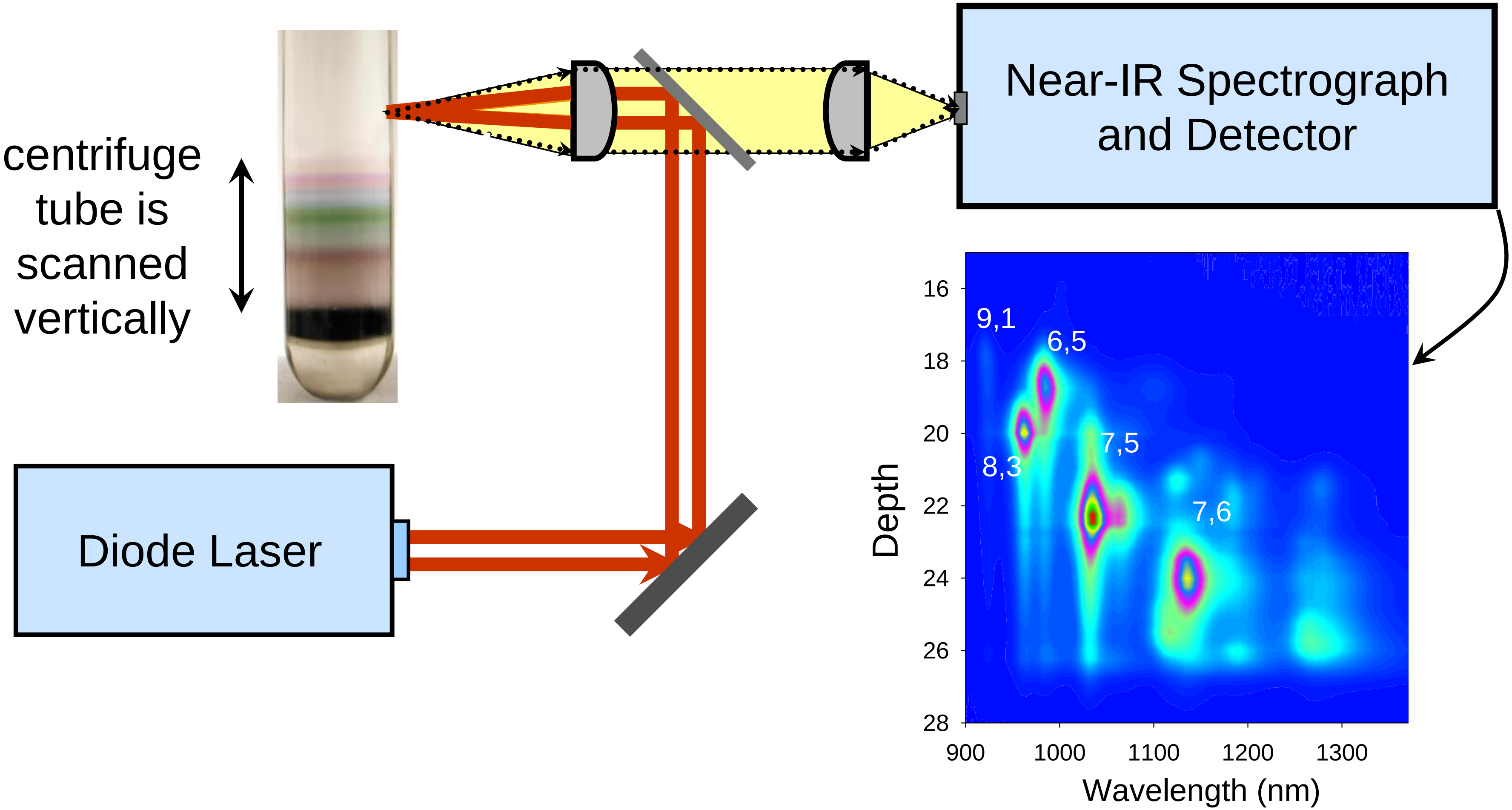
Optional vertical sample scanning gives *in situ* fluorescence maps of SWCNTs separated using centrifuge techniques



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NanoSpectralyzer Benefits

- Unique multimode spectrometer designed for nanomaterial analysis, providing
  - Near-infrared fluorescence
  - Near-infrared absorption
  - Visible fluorescence
  - Visible absorption
  - UV absorption
  - Raman with one or two excitation wavelengths
- Highest sensitivity and speed
- Automated interpretation of NIR SWCNT fluorescence spectra
- Automated measurement of emission efficiency
- Compact tabletop instrument
- No liquid nitrogen use; modest power consumption
- Turn-key operation for research or routine applications
- Designed and built by scientists for scientists

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Training PowerPoint

Applied NanoFluorescence, LLC

2018

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