

## Better Data on Carbon Nanotubes with the NanoLog

FL-29

ELEMENTAL ANALYSIS

FLUORESCENCE

GRATINGS & OEM SPECTROMETERS

OPTICAL COMPONENTS

FORENSICS

PARTICLE CHARACTERIZATION

R A M A N

SPECTROSCOPIC ELLIPSOMETRY

SPR IMAGING

### Introduction

Improvements to the HORIBA Scientific NanoLog® (Fig. 1), already the best spectrofluorometer for exploration of single-walled carbon nanotubes (SWCNTs), render it even more suitable for this application. We have added absorbance measurement capability and increased sensitivity by over five times. Because SWCNT length is directly proportional to absorbance (A) and photoluminescence (PL) intensity, you can now better understand the nature of your SWCNT samples. The NanoLog® can provide data on metallic SWCNTs, plus determine concentrations of semi-conducting SWCNTs. In addition, the increased sensitivity of the NanoLog® permits acquiring a complete PL excitation-emission matrix (EEM) in < 3 min.



Fig. 1. NanoLog® system.

### Sample absorbance measurements

Excitation bandpass = 5 nm, with 0.1 s integration time per 1 nm increment. The excitation grating was 1200 grooves  $\text{mm}^{-1}$ , blazed at 330 nm. The detector was a Si-PIN diode at room temperature. The SWCNT sample<sup>1</sup> was placed into a rectangular cuvette with 0.5-cm path-length, and diluted to  $A \approx 0.09$  at 981 nm (Fig. 2).

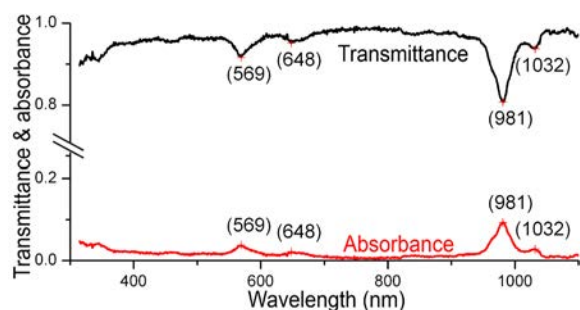


Fig. 2. Absorbance (red) and transmittance (black) of NIST SWCNTs, using the new absorbance function of the NanoLog®. Important peaks are labeled.

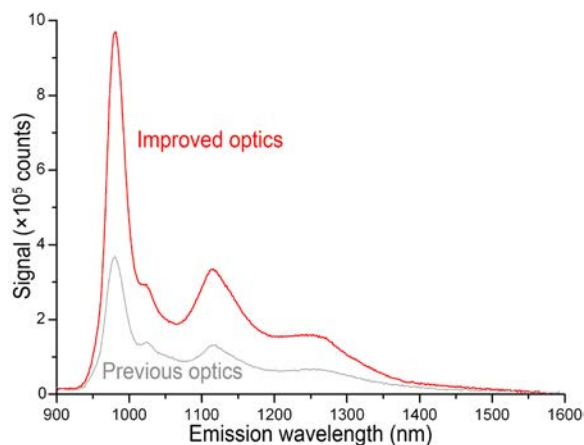
### Increased sensitivity and higher S/N

To demonstrate the NanoLog®'s improved sensitivity, (6,5) SG SWCNT (CoMoCAT method<sup>3</sup>) was used ( $0.1 \text{ mg L}^{-1}$  in  $\text{D}_2\text{O}$ , with 0.1% NaDDBs surfactant). The sample was put into a 1-cm path-length cuvette inside the NanoLog® to measure absorbance at  $90^\circ$  to incident light. The NanoLog® used a 1" InGaAs  $512 \times 1$  array detector,  $\text{N}_2(l)$ -cooled to 170 K, and a Schott RG830 cut-on filter ( $\lambda > 830 \text{ nm}$ ) in the emission path. With 10 nm bandpass and  $2 \times 5 \text{ s} = 10 \text{ s}$  integration time, the scan was centered

<sup>1</sup> Fraction 9, J.A. Fagan, et al., "Length-Dependent Optical Effects in Single-Wall Carbon Nanotubes", J. Am. Chem. Soc. 129(34), 2007, 10607–10612.

<sup>3</sup> SouthWest NanoTechnologies, Inc., 2501 Technology Place, Norman, OK 73071-1102

at 1210 nm. The excitation monochromator grating was the same. The emission spectrometer grating was 100 grooves mm<sup>-1</sup>, blazed at 800 nm. The new absorption accessory gave A = 0.72 at 982 nm. PL from the sample was scanned at  $\lambda_{exc} = 568$  nm. Signal (Fig. 3) from the improved NanoLog® (red) is three times higher than the original system (gray).



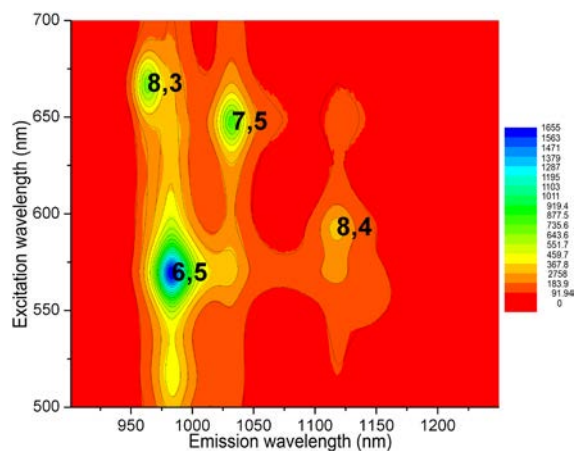
**Fig. 3.** PL signal from the CoMoCat sample described above. Gray: original NanoLog® system; red: improved instrument.

S/N determination was similar to the previous experiment, with the same detector, cut-on filter, center wavelength, optical path, bandpass, integration time, and gratings. The SWCNT sample<sup>1</sup>, in a 0.5-cm path-length rectangular cuvette, was diluted to A ≈ 0.09 and scanned from 828–1520 nm. Calculated<sup>4</sup> S/N = 19 200 from the (6,5) peak and average blank signals. An EEM was created via the NanoLog® and its Nanosizer software (Fig. 4) with prominent peaks (>10% of maximum) labeled. The Nanosizer software also produced a helix-angle map of the sample's components (Fig. 5).

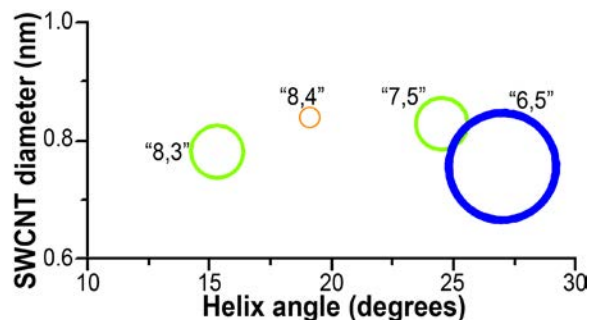
### Conclusions

The new absorbance accessory and upgraded optics have improved the HORIBA Scientific NanoLog® spectrofluorometer, yielding more complete characterization combined with significantly faster throughput, more signal, and higher S/N for SWCNT experiments.

<sup>4</sup> Peak signal = 78 933, average blank signal from 828–1520 nm) = 16.8.



**Fig. 4.** EEM of NIST sample, with important SWCNTs (>10% of maximum) labeled using (n,m) coordinates.



**Fig. 5.** NIST SWCNT diameters vs. helix-angle. Circles' diameters  $\propto$  intensities in Fig. 4.