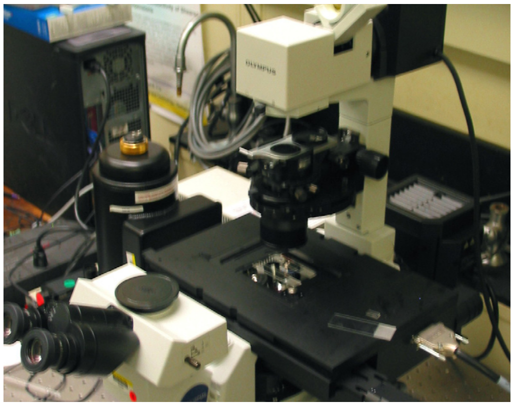
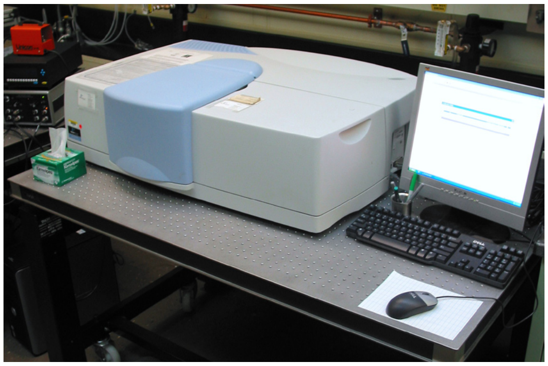
Measurement Facilities:
NIST Combinatorial Methods Center



Microscope capable of imaging individual SWCNTs in the near infrared.

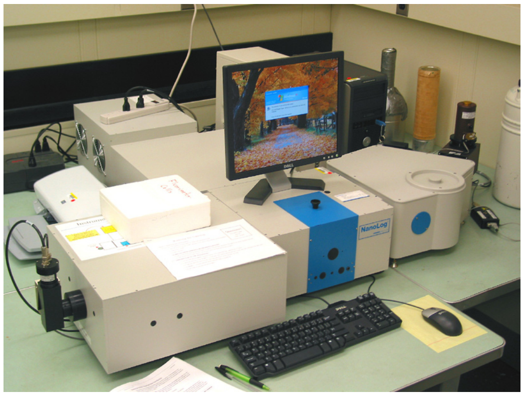
Critical optical and electrical SWCNT properties are measured in MSEL's Polymers Division using a variety of instruments. Optical absorption in the Ultraviolet-Visible-Near Infrared range can be measured in either transmission or reflectance modes from (185 to 3300) nm with a sensitivity of below 0.001 absorbance units. UV-Vis-NIR thus allows for rapid deter-mination of concentration and the rough distribution of chiralities in either liquid or transparent solid samples. Rapid integration times further allow for the study of time-dependent phenomena.



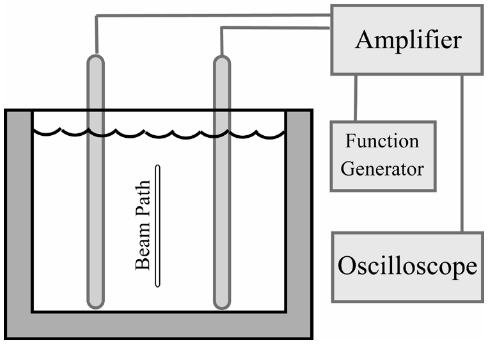
Ultraviolet-Visible-Near Infrared range instrumentation.

Near Infrared Fluorescence allows for a more accurate assessment of the distribution of semiconducting SWCNTs in a liquid sample, and can be measured for an excitation wavelength range of 220 nm to 1 μm with fluorescence detection ranging from (290 to 1700) nm.

In addition to these nanotube facilities in the Polymers Division, we collaborate with groups engaged in Raman spectroscopy, small-angle scattering, and TGA.



Fluorimeter for identification of semiconducting SWNT chirality.



Barry Bauer
(301) 975-6849
barry.bauer@.nist.gov

Jeffrey Fagan
(301) 975-6740
jeffery.fagan@.nist.gov

Erik Hobbie
(301) 975-6774
erik.hobbie@.nist.gov

Kalman Migler
(301) 975-4876
kalman.migler@.nist.gov

Measurement Facilities:
Carbon Nanotube Separations

The Polymers Division has established facilities capable of purifying and sorting single wall carbon nanotubes (SWCNTs). Size exclusion chromatography (SEC), field flow fractionation (FFF), ion exchange chromatography (IEC), and ultracentrifugation (UC) instrumentation have been specifically designed to sort SWCNTs by length and chirality. Property measurements of SWCNT fractions are initially conducted on-line during the fractionation of liquid dispersions. Fractions are collected for measurements by other facilities to characterize purity, size, mobility, optical and electrical properties.

Chromatographic Separations

Our liquid chromatographic facility is made of an extensive combination of pumps, separation components, and on-line detection. Pumps produce the stable constant flow rate necessary for SEC and associated detection. They are also programmable for the flow rate changes necessary for FFF and mobile phase composition changes necessary for IEC. Separations are made by nano-porous SEC or IEC columns or by controlled fields in a FFF channel. On-line detection includes size sensitive instrumentation such as viscometry and multi-angle light scattering and chirality sensitive instrumentation such as a photo diode array for absorption spectroscopy.

Top: A liquid chromatographic system has been designed and constructed to separate SWCNTs by SEC, FFF, and IEC.

Bottom: FFF employs a complex flows to separate by size.

Ultracentrifugation

Our ultracentrifuge, and the correct choice from several sizes and types of rotors, provides the force necessary for separations by size or chirality of SWCNTs through either kinetic or equilibrium approaches. Centripetal acceleration of over 400,000 g can be applied in the fastest rotor.

Batches of up to 0.5 L of SWCNT dispersion can be separated in the largest rotor, processing up to 20 mg of SWCNT per run, or down to 5 mL in the smallest rotor, allowing for multiple parallel experiments. Although online characterization is not available, an available fractionator allows for the continous extraction of fluid from the processed cells, which can be fed into the SEC-FFF instrumentation.

Ultracentrifugation facility is capable of separation by size or chirality.

Optical and Electrical Measurements

Near infrared fluorescence microscopy instrumentation exploits the intrinsic photo luminescence of semi-conducting single-wall carbon nanotubes in the near infrared to image individual nanotubes in real time. A broad-band optical excitation source or a laser is used to excite the higher-order optical resonances of semiconducting nanotubes. A dichroic mirror with a long-pass optical filter is used to collect the near-infrared emission onto a liquid nitrogen cooled InGaAs CCD camera. Individual nanotubes of length 500 nm or greater can be easily imaged with this technique and diffusivity can be measured.

FACILITIES