# Workshop Summary

## Fourth NIST Workshop on Carbon Nanotubes: Chirality Measurements

Organizing Committee
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On September 23rd and 24th 2010, the National Institute of Standards and Technology (NIST) hosted the fourth in a series of workshops addressing measurement needs for single wall carbon nanotubes (SWCNTs). Attendees representing an international community, including members of industry, academia, government laboratories, and regulatory agencies, participated in the workshop. The primary focus of this workshop was on SWCNT chirality population measurements and chirality-dependent properties.

The workshop consisted of three sessions. The first morning was devoted to presentations and discussion of an inter-laboratory study on various methods for determination of the chirality distribution of SWCNTs, which was conducted under the auspices of the Versailles Project on Advanced Measurements and Standards (VAMAS). Such measurements are critical for specifying the properties that can be expected from and the purity of nanotubes for commerce. These methods included tip-enhanced Raman spectroscopy, fluorimetric analysis, Raman spectroscopy, and transmission electron microscopy. The material used in the inter-laboratory study was the *Long* fraction of a NIST reference material that will be publicly available for all purchasers to compare against (RM 8281, length-separated SWCNT populations). So far, 12 of the laboratories, representing seven countries, participating in this round robin have submitted results.

The early results of the inter-laboratory study show dramatically how different techniques and their level of difficulty affect the measured population distribution. The advantages and disadvantages of various techniques imply that continued caution is needed in interpreting population data. The results of the study will be published and used to enable techniques for the absolute determination of the SWCNT chiral vector distribution in a dispersed population.

The second session focused on techniques for chirality separation and selective synthesis. Most of these presentations dealt with advances in the methods for separating nanotube populations. Highlights were detailed discussion of three different techniques: density gradient ultracentrifugation (DGU), agarose gel column chromatography, and ion-exchange chromatography using sequence-selective DNA wrapping of the nanotubes. Specifically, Mark Hersam described advances in density gradient centrifugation that could be used to create samples of 98 % to 99 % pure SWCNT populations. He additionally discussed a number of new nanotube-based devices that could result from this separation technique. Hiromichi Kataura presented a multi-stage chirality separation via a gel-filtration method that enables sequential isolation of all the small-diameter semiconducting chiralities in a HiPco sample. Xiaomin Tu presented the technique of sequence-selective DNA-based chirality separation, and new work supporting the DNA structure – selectivity hypothesis. As presented by Nathan Yoder of Nanointegris corp., the cost of separated SWCNTs, and ultimately the cost of any application derived from these nanotubes, is critically dependent on the ability of the separation technologies to produce them in the needed chiralities and sizes in sufficiently large quantities.

Separately, R. Mohan Sankaran and Sivaram Arepalli both talked about methods for influencing the distribution of synthesized nanotubes. These included control of the catalyst size and synthesis conditions. Results presented showed that these variables can shift the synthesized population of nanotubes, and may lessen the need for post-production separation techniques.

The final session was devoted to applications that are enabled by chirality-separated SWCNT populations. These included field-effect transistors, thin-film solar devices (Jeffrey Blackburn and Michael Arnold), and nano-therapeutics and combined targeting / bioimaging agents (Hongjie Die and Shawn Chen). It was also noted that the purification of single nanotube species is allowing the study of previously intractable photophysical measurements (Stephen Doorn).

The talks on the second day also acknowledged a reality brought up in the earlier session: Although there have been significant advances in separation technologies for large-scale production of different nanotube populations, significant efforts toward rapid quality control are still necessary to commercialize advanced applications. Similarly, there was discussion about the need for improved capabilities for specification of the chiral purity in samples for various electronic applications. There was a consensus that ability to specify the semiconducting purity to several decimal places is needed, with five 9's (99.999 %) or more being desirable. However, the difficulties that have been encountered in this specification are not just in the removal of intransigent impurity species, but also the missing ability to rapidly screen materials and identify contaminating species. A lack of tools was specifically identified for separation of chiralities with diameters larger than 1 nm.

## Conclusions

Although only a few short years have passed since the previous nanotube workshop at NIST, tremendous progress has been made in the separation and utilization of different SWCNT chiralities and populations. Despite the economic climate, industrial production and use of purified and controlled nanotube populations has emerged to bridge the gap between material science and applied products.

The participants in this workshop were uniformly enthusiastic about nanostructured carbon materials and share the opinion that chiral separation techniques and measurement procedures are important areas for continuing work. Although participants were excited about the progress in isolation of specific nanotube functionalities, the continuing obstacle to the development of many nanotube-based electronics and the highest technological applications is the limited availability of single-chirality SWCNTs in sufficient volumes.

The exciting progress of nanotube science and technologies in the last few years has not eliminated the need for advanced measurement science, but is pushing it in new directions for more advanced control and rapid screening, that will result in new capabilities and applications of nanotube materials.