



**Peer Consultation Public Meeting on
The Material Characterization of Nanoscale
Materials**

Discussion Topics

August 7, 2007

August 07, 2007

**Peer Consultation Public Meeting on
The Material Characterization of Nanoscale Materials**
Discussion Topics

Meeting Background and Purpose

EPA is convening a public scientific peer consultation meeting on material characterization for nanoscale chemical substances (“nanoscale materials”) to inform the development of its Nanoscale Materials Stewardship Program (NMSP) under the Toxic Substances Control Act (TSCA). The peer consultation is one of several actions EPA is taking to better understand the potential risks and benefits of nanotechnology.

On October, 18, 2006, EPA invited the public, industry, environmental groups, other federal agencies and other stakeholders to participate in the design, development and implementation of a stewardship program for nanoscale materials. On July 12, 2007, EPA announced the availability of a NMSP concept paper and related documents and a public meeting on August 2, 2007 to discuss and receive comments on these materials (<http://www.epa.gov/oppt/nano/nmspfr.htm>).

The NMSP will complement and support the Agency's new and existing chemical programs under TSCA and will help provide a firmer scientific foundation for regulatory decisions by encouraging the development of key scientific information and appropriate risk management practices for new and existing chemical nanoscale materials. The NMSP is intended to include but not limited to engineered nanoscale materials manufactured or imported for commercial purposes as defined in 40 CFR 720.3 (r). The NMSP is envisioned to have two parts: 1) a Basic Reporting Program and 2) an In-depth Program. This discussion paper and the public peer consultation meeting are intended to support the NMSP by identifying material characterization data that participants could submit under the Basic Program if they are in the participant's possession or are reasonably ascertainable as defined in 40 CFR 720.3 (p). The data and experience generated by the basic reporting phase will help to inform the types of in-depth data to be developed. In-depth data development could begin at any time and would entail, among other types of data, development of material characterization data in a greater amount of detail. In-depth data development could also include additional types of material characterization data if they are identified.

EPA received input in November, 2005 from the National Pollution Prevention and Toxics Advisory Committee (NPPTAC) regarding a voluntary stewardship program for nanoscale materials. A NPPTAC ad-hoc work group on nanoscale materials developed an overview paper that stated that a voluntary stewardship program should:

1. Give EPA, and the public to the extent possible recognizing legitimate CBI issues, a better understanding of the types of engineered nanoscale materials; the physical, chemical, hazard and exposure characteristics of such substances; the volume of such substances; and the uses of such substances;

2. Help EPA develop capacity and a process to identify and assess risks of engineered nanoscale materials;
3. Help EPA determine what information it needs about engineered nanoscale materials and articulate those information needs to industry and other stakeholder groups;
4. Help EPA understand what risk management practices are being used at production, processing, use and disposal stages, and what additional risk management practices need to be implemented;
5. Prompt or reinforce the implementation of risk management practices; and
6. Provide the information and experience needed to develop an overall approach to the treatment of nanoscale chemical substances under TSCA that builds public trust in nanoscale materials while enabling innovation and responsible development.

EPA will utilize all public input, including that from NPPTAC, other stakeholders, public meetings and peer consultations to further inform the development of its Nanomaterials Stewardship Program and TSCA program for nanoscale materials.

Meeting Objectives

The EPA public peer consultation meeting on material characterization needs for nanoscale materials will help clarify which data and elements should be included in the NMSP Basic Program and/or In-depth Program. The goal is to have an applied discussion that considers 1) the currently available understanding of material characterization as it relates to nanoscale materials and 2) how this understanding can be used to guide the Agency's thinking regarding the material characterization data elements that would be most useful and important to include in the NMSP. The specific objectives of the public peer consultation meeting are as follows:

1. To inform industry and the public of EPA's level of understanding of the material characterization needs for nanoscale materials in general and for the NMSP;
2. To further develop EPA's understanding of how nanoscale materials are engineered or manufactured to achieve specific properties and characteristics;
3. To further develop EPA's understanding of which chemical identification elements and physical-chemical property data are generally relevant in characterizing nanoscale materials and which identification elements and property data are most important in characterizing specific classes of nanoscale materials;
4. To discuss what analytical procedures and test methods are available for acquiring these material characterization data, and where procedure and method validation or development is needed;
5. To discuss how these material characterization data needs should be prioritized for the NMSP Basic and/or In-depth Program;

Discussion Overview

Despite the rapid advancement of nanotechnology, the breadth of nanoscale material types coupled with the limited hazard data available for many of these materials pose a challenge in understanding and measuring their benefits and risks. Numerous efforts are underway to begin to address these challenges. The International Life Sciences Institute (ILSI) Research Foundation/Risk Science Institute, for example, convened an expert working group to develop a strategy for identifying hazards associated with engineered nanoscale materials. Focusing on the limited data available, the working group developed a screening strategy for hazard identification (rather than a detailed testing protocol) that includes a broad data gathering effort.¹

The ILSI report describes the characterization of nanoscale materials, in addition to in vitro and in vivo screening, as a key third aspect of an overall screening strategy due to the likely dependence of the biological activity of nanoscale materials on physical-chemical properties not often considered in toxicity screening studies. Additionally, given the difficulties associated with characterizing many nanoscale materials, nanoscale material characterization is a subject appropriate for detailed investigation and discussion.

Numerous national and international standards organizations, including the American National Standards Institute (ANSI)² and the International Organization for Standardization (ISO)³, have also convened committees to begin to address many of these same challenges and in particular the need for methods standardization. Several of these committees have indicated that, given the breadth of nanotechnology and its data issues, the initial products the committees develop may be in the form of best practices rather than actual test protocols.

The remainder of this paper discusses a proposed approach for the EPA scientific peer consultation meeting on nanoscale material characterization. EPA recognizes that the different chemical classes of nanoscale materials would make universal application of any particular characterization endpoint or methodology impossible. The premise of the Basic Reporting phase of the NMSP is that some information is known or reasonably ascertainable. The approach for this panel is therefore to discuss what data are known or reasonably ascertainable to characterize nanoscale materials. This will be followed by a discussion on the methodology used to obtain and use characterization endpoints of interest.

Discussion Topics

I. Characterization of Nanoscale Materials

1. Description of nanoscale materials

a. Types/categories of nanoscale materials

¹ [http://www.particleand fibretoxicology.com/content/2/1/8](http://www.particleandfibretoxicology.com/content/2/1/8)

² http://www.ansi.org/standards_activities/standards_boards_panels/nsp/overview.aspx?menuid=3

³ <http://www.iso.org/iso/en/stdsdevelopment/tc/tclist/TechnicalCommitteeDetailPage.TechnicalCommitteeDetail?COMMID=5932>

2. Physical-chemical properties of potential interest
 - a. Particle size and distribution
 - b. Particle shape and dimensions
 - c. Agglomeration and aggregation
 - d. Surface area
 - e. Surface charge
 - f. Surface chemistry
 - g. Chemical composition
 - h. Crystal structure
 - i. Impurity identification and levels
3. Design to achieve unique properties
 - a. Manufacturing and processing methodologies
 - b. Chemical transformations

II. Methodologies for Characterizing Nanoscale Materials

1. Obtaining characterization data for nanoscale materials
 - a. Analytical methods for detecting and quantifying nanoscale materials
 - b. Analytical methods measuring physical-chemical properties (measurement techniques and testing protocols)
 - c. Models to predict properties and effects
2. Metrology
 - a. Methods validation
 - b. Standards and harmonization
3. Prioritization of characterization data and data gaps
4. Miscellaneous – Do Panel members have additional topics to discuss?

Discussion

This section provides additional information on specific technical issues to facilitate discussion at the meeting. The information will include literature findings as well as questions on the specific discussion topics.

Types of Nanoscale Materials & Their Structures and Chemical Compositions

Based on structure and chemical composition, EPA has grouped nanomaterials into 4 distinct categories for purposes of this discussion: 1) simple organic molecules; 2) simple inorganic molecules; 3) polymeric substance (including dendrimeric substances); and 4) composites. A fifth category, biological compounds will not be addressed in the peer

consultation. While all of these categories can be divided further, only the organic category will be divided further into molecules based predominantly on carbon (e.g., fullerenes, nanotubes) and all other organic substances (e.g., salts of carboxylic acids). This grouping is similar to the American National Standards Institute Nanotechnology Standards Panel approach presented at the September 2004 meeting at the National Institute of Standards and Technology in Gaithersburg Maryland⁴.

Question 1: Are there any other significant categories, based on structure and chemical composition, that should be included in this discussion because they are substantially different from the categories mentioned (e.g., hybrids, self-assembly devices, others)?

Question 2: For the different categories of nanoscale materials, what is the current state of knowledge about structure and chemical composition?

Question 3: Can structures and chemical composition be correlated to specific properties and is this correlation quantifiable?

Physical-Chemical Properties

The importance of nanoscale materials is due to their potential for unique or greatly enhanced properties. EPA routinely uses a base set of physical-chemical property data (e.g., melting point, boiling point, vapor pressure, water solubility) for a variety of programs (e.g., High Production Volume Challenge, New Chemicals) and decision-making. As mentioned previously, certain material properties are of significant importance in characterizing nanoscale materials. Recent research suggests that particle size, surface area, and surface chemistry (or surface activity) are initially some of the most important properties to measure.

As expected for most chemicals, class 1⁵ substances having specific molecular structures and formulas may be more readily studied and characterized at the nanoscale than the polymer and composite categories. For example, carbon-based nanoscale materials as well as metal oxide nanoscale chemicals are often well characterized. Structural and physical-chemical property data therefore are likely to be well documented for these types of materials.

Question 4: Which physical-chemical properties are relevant to nanoscale materials and how? Which are known or reasonably ascertainable and which have data gaps?

Question 5: Are there properties that would have little or no relevance under the NMSP?

Question 6: Which properties are associated with aggregated or agglomerated nanoscale materials, as opposed to properties that are inherent to the material regardless of physical form?

⁴ <http://publicaa.ansi.org/sites/apdl/Documents/Forms/AllItems.aspx>

⁵ Class 1 substances are distinct chemicals with known, non-variable molecular structures

Question 7: Are there routine manipulations of nanoscale materials that result in physical-chemical properties changes or other defining characteristics (e.g., surface modifications of nanotubes to enhance solvent dispersibility)?

Question 8: How should physical-chemical property data be prioritized for the NMSP? Based on availability, effect (toxicity or exposure criteria), or other factors?

Nanoscale manufacturing and processing

The number of manufacturing and processing methods for generating nanoscale material continues to grow and become more sophisticated. The two primary areas for this discussion include physical reduction methods (milling) and engineering methods (e.g., particle stabilization, vapor deposition, self assembly).

Question 9: What are the common processes used to manufacture nanoscale materials?

Question 10: How are processes used to produce specific characteristics or properties?

Question 11: Which methods reduce particle size but do not result in property changes? Which methods reduce particle size and result in property changes?

Impurities

Impurity content is a growing area of interest in nanotechnology due to improved performance observed in some cases (e.g., solar cells and semiconductors) and deleterious effects observed in others (e.g., quantum dot quantum computers). The confounding effects that impurities have with respect to toxicological endpoints is also being studied (National Nanotechnology Initiative 2006 Environmental Health and Safety research report⁶).

Question 12: How important are impurity identity and impurity levels to the understanding and characterization of nanoscale materials?

Question 13: Are there routine purification procedures that can effectively control or remove impurities, when desirable, for certain classes of nanoscale materials?

Obtaining characterization data

Determining identity, quantifying the nanoscale particle range, and measuring physical-chemical properties for that identity and particle range are essential to the characterization of nanoscale materials. Because of the challenges associated with size, shape, surface characteristics, and possibly other aspects of nanoscale materials, an evaluation of existing measurement techniques is critical to nanoscale material characterization. The National

⁶ http://www.nano.gov/NNI_EHS_research_needs.pdf

Nanotechnology Initiative report stated that “...Accurate and useful measurement techniques are also important because agglomerated nano materials may either retain or lose their emergent properties - or take on new properties - thus affecting the potential biological response.”

Question 18: Are validated methods available for the different categories of nanoscale materials?

Question 17: Are there techniques that can be universally applied?

Question 19: For small quantities of materials, are there sampling, handling, and collection techniques as well as sample integrity, accuracy and precision QA/QC methodologies available?

Question 20: What is the status of standardization efforts? Are these efforts focused on broadly applicable characterization methods or category-specific methods?

Question 21: What alternative or innovative methods or technologies can be applied to nanomaterial analysis?

Modeling

Empirical modeling can be a useful approach to predict physical-chemical properties when experimental data are not known or ascertainable. The initial problem with modeling is that, to accurately predict property endpoints for a given category of substances, there must be some experimental data available in the tool's database for at least some representative substances in that category. For newly discovered or studied materials, the minimum but necessary quantity and type of experimental data often is not available to sufficiently populate a tool's database and allow accurate prediction by the tool. Some estimation methods have been developed for specific property endpoints, but many others are lacking.

Question 22: Are there models that are currently used to obtain property data for nanoscale materials? For which properties and which nanoscale materials?

Question 23: Has any validation work been conducted that compares predicted values with measured data? For which properties and which nanoscale materials?

Question 24: Are there current significant characterization needs for which the NMSP should investigate model development?