



# Nanomaterials and the Environment & Instrumentation, Metrology, and Analytical Methods

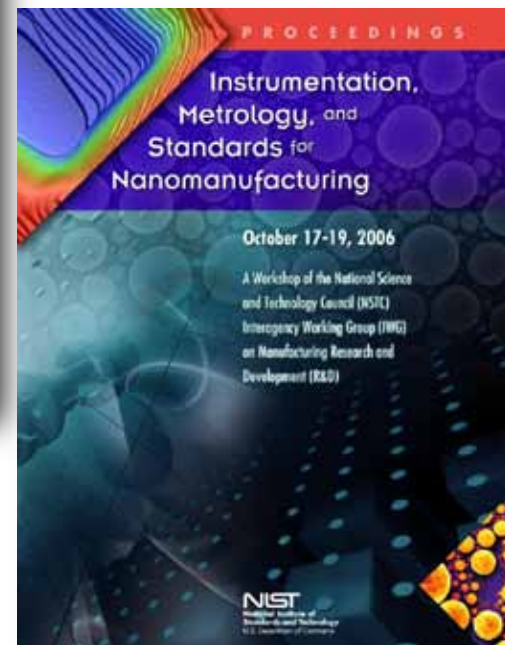
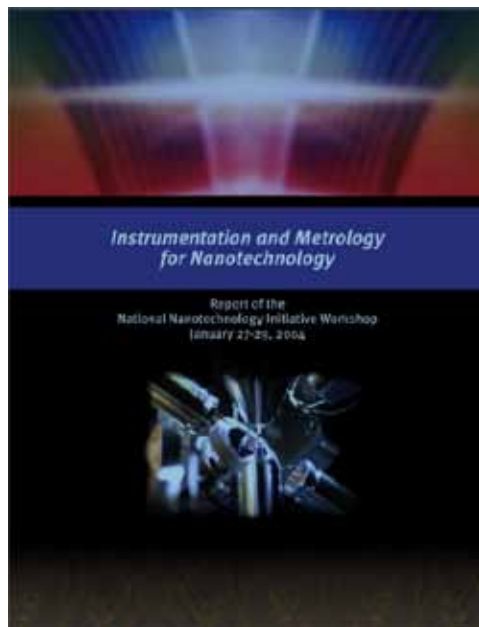
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*Measuring and predicting levels of exposure of nanomaterials for various species in the environment*

- **National Institute of Standards is the National Measurement Laboratory**
  - Precision Engineering Division has the responsibility of length measurement and traceability to the International System of Units (SI) – meter.
  - Responsibility extends over 12 orders of magnitude from kilometers to nanometers
- **Subject matter expert comments are for measurement of length – size and dimensionality**
  - Infrastructural measurements
  - Many of the comments can be broadly applied.

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- Two previous NNI Workshops spoke directly to the instrumentation, metrology and analytical methods needed to broadly measure nanomaterials.



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## Earlier Workshops

- Much of the measurement infrastructure currently available for nanoparticle metrology good and getting better but, progress is only incremental in nature:
  - Optics
  - Transmission Electron Microscope
  - Scanning Probe Microscope
  - Scanning Electron Microscope
- Automated, operator-independent instrumentation adapted to nanomanufacturing must be developed and is indispensable to future manufacturing
- New, potentially revolutionary metrology and measurement tools are needed for many applications
  - Helium Ion Microscope



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- Over the past 20+ years, the semiconductor industry has been the main financial and technical driver for revolutionary advances in many areas of micro and nanomanufacturing systems and processes.
  - Resulted in smaller, faster and less expensive devices.
    - Common to go to the computer store and obtain microprocessors with 45 nanometer and smaller technology
- We should look to that industry for a successful model for development of the needed infrastructural instrumentation for measurement of small structures – nanomaterials in the environment

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The evolution of microelectronics to nanoelectronics, following Moore's Law, has been facilitated through:

- Industrial consortia such as SEMATECH
- The Semiconductor Industry Association (SIA)
- SEMI
- International Technology Roadmap for Semiconductors (ITRS)



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- **NNI Workshops concluded that**
  - **A coordinating consortium-type organization (or organizations) could make huge strides in the development of needed instrumentation and metrology**
    - Reduce duplication of effort
    - Focus resources
  - **Nanotechnological commonalities of need in instrumentation, metrology and standards must be identified and a focus must be developed**
    - Act as the focal point for any road mapping exercises.
- **The “Bible” of the semiconductor industry has been the International Technology Roadmap for Semiconductors**

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- The ITRS has successfully guided:
  - General technology development
  - Instrument manufacturers guidance to provide the needed tools with reasonable lead time.
- Demanding industry that has continually pushed the capabilities to measure small structures
  - Were able to do this because of the wealth of the industry
- Instrument development associated with the semiconductor manufacturing industry was/is an evolutionary process
  - Fueled by the defined needs of the ITRS and funded by the established semiconductor industry.
- Instruments for measurement of nanomaterials especially those in the environment (nanoparticles) will likely follow a similar path
  - But, the manufacturers need credible guidance in order to risk investment
- Today, the emerging nanomanufacturing industry does not have sufficiently deep pockets to fund similar high risk instrument development.
  - Potentially creates a significant funding gap leading to an impending technology gap.



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## Accuracy vs. Precision

- Most instruments can demonstrate a high degree of precision, but are they accurate?
- Size matters where nanotechnology is concerned
  - Knowing dimension with a known uncertainty is primary to understanding the function of nanomaterials
  - Measurements rely on instrument response functions – varies substantially between instruments
  - Often more complex than realized and is why different instruments give different measurements
    - Scanning electron microscope – electron beam interactions dominate
- Many of the databases currently under development are incorporating data from multiple sources.
  - Are these sources accurate in the generation of these data?
  - Nanomaterial database development relies on the quality and consistency of the data submitted.
  - In order to describe the properties of a nanomaterial, accurate measurement infrastructure is needed and adopted so that these data are valid and useful.
  - Data is being generated for:
    - Nano-risk Framework (DuPont and Environmental Defense Fund),
    - NIH NanoHealth Initiative,
    - EPA – Nanoscale Materials Stewardship Program and others
- Interlaboratory studies having well defined measurement procedures using accurate samples are critical to achieving confidence in the overall data.

## Accuracy vs. Precision

- The need for accurate measurements is greater than ever because there is so little tolerance for error
  - Accuracy is telling the truth (within some measurement uncertainty)
  - If accuracy is not assured precision is telling the same lie over and over again.
- The need for clear, concise standardized assessment of shape and surface area requires much attention
  - Consistent measurement algorithms

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## Accuracy vs. Precision

– NIST is working with the other agencies, industry and academia to develop the needed metrology and standards. For example:

- NIST/FDA/NCI
  - Three gold nanoparticle standards (RM 8011, RM 8012, and RM 8013)
  - (others in the pipeline)
- NIST/NIOSH/RJ Lee Associates
  - Carbon nanotube metrology

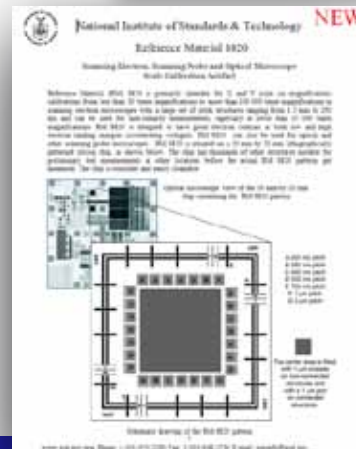


– NIST/SEMATECH

- Accurate scanning electron microscope calibration standards

– Accurate standards require:

- Quantity of high-quality relevant material
- Homogeneous material
- Needed metrology
- Accurate measurement instrument
- Determine the total uncertainty of the measurement



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- In the real world materials do not always cooperate:
  - Carbon nanotube – two words for many different possible materials
  - At least 50 different CNT species have been identified
  - Only half of these species are semiconducting
  - Current manufacturing processes do not simply make one type of CNT
    - Inherently produce a mixture of CNT species along with 3-60%+ unwanted chemical impurities
  - Early studies have found that residual catalyst and other contaminants can make the material inhomogeneous.



MWCNTs 270 tons/yr (245 000 kg/year)  
SWCNTs 7 tons/yr (6 350 kg/year)  
8 000 US\$/kg to 500 000 US\$/kg

<http://www.wtec.org/cnm/>

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## Modeling is Critical

- With the large amount of potential nanoparticles and the nanoparticle combinations possible, predictive modeling will become crucial.
  - [Workshop on Cross Industry Issues for Nanomanufacturing](#)
  - [Predicting exposure limits and effects](#)
- Model development is only as good as the input data
- Data needs to be accurate so informed decisions can be made regarding the suitability of these materials as a commercial product, a component of a commercial product, or release into the environment are made on a strong *scientific* basis and not hype.

## Final Comments:

- *If a nanomaterial cannot be measured it cannot be manufactured.*
- *If a nanomaterial cannot be made safely it should not be manufactured.*
- *If a nanomaterial cannot be measured how would you even know?*

Thank you

NLST

HV

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# Contact Information:

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- Instrumentation and Metrology for Nanotechnology  
[http://www.nano.gov/NNI\\_Instrumentation\\_Metrology\\_rpt.pdf](http://www.nano.gov/NNI_Instrumentation_Metrology_rpt.pdf)
- Instrumentation, Metrology and Standards for Nanomanufacturing <http://www.manufacturing.gov>
- Cross Industry Issues for Nanomanufacturing (in preparation)



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## Back up slides

# Gold Nanoparticle Size Standard RM

RM 8011 - 10 nm

RM 8012 - 30 nm

RM 8013 - 60 nm



National Institute of Standards & Technology

## Report of Investigation

Reference Material 8011

Gold Nanoparticles, Nominal 10 nm Diameter

This Reference Material (RM) is intended primarily to evaluate and qualify methodology and/or instrument performance related to the physical/dimensional characterization of nanoscale particles used in pre-clinical biomedical research. The RM may also be useful in the development and evaluation of *in vitro* assays designed to assess the biological response (e.g., cytotoxicity, hemolysis) of nanomaterials, and for use in interlaboratory test comparisons. RM 8011 consists of nominally 5 mL of citrate-stabilized Au nanoparticles in an aqueous suspension, supplied in hermetically sealed pre-sterilized glass ampoules sterilized by gamma irradiation. A unit consists of two 5 mL ampoules. The suspension contains primary particles (monomers) and a small percentage of clusters of primary particles.

**Expiration of Material:** The reference values for RM 8011 are valid, within the measurement uncertainties in RM is handled in accordance with the instructions given in this report. The size distribution may be altered and the RM invalidated if the

monitor representative samples from this RM lot over the period of the reference values before the expiration date. NIST will notify you if facilitate notification.

preparation, processing and measurement activities was conducted in the

tests were performed at NIST by the following: NIST Analytical Chemistry Division: L.C. Sander and M.R. Winchester; NIST Ceramics Division: D.-I. Kim and P. Nambudiri; NIST Metallurgy Division: M.L. Becker, D.L. Ho, A. Karim and B.M. Vogel; NIST U.S. VLSI Division: M.L. Becker, D.L. Ho, A. Karim and B.M. Vogel; NIST U.S. VLSI Division: M.L. Becker, D.L. Ho, A. Karim and B.M. Vogel; NIST U.S. VLSI Division: M.L. Becker, D.L. Ho, A. Karim and B.M. Vogel.

and analysis of the reference value data were performed by the

were provided by the following: R.F. Cook, W.K. Butler and

and issuance of this RM were coordinated through the NIST

National Cancer Institute (NCI). Development and production costs

Debra L. Kaiser, Chief  
Ceramics Division

Robert L. Watkins, Jr., Chief  
Measurement Services Division



RM 8011

Report of Investigation available at:

<http://ts.nist.gov/measurementservices/referencematerials/index.cfm>

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- The NIST Manufacturing Engineering Laboratory (MEL) has been supporting nanomanufacturing through the development of programs for measurements and standards since about 1999.
  - MEL plays a leading role in developing Standard Reference Materials (SRM), metrology and measurement instrumentation.
- Semiconductor manufacturing has clearly benefitted and has demonstrated the value of this work
- RTI International Report estimates that for every \$1 spent on measurement, the industry as a whole saw a \$3.30 return.
- Metrology is and has proven to be value added.

