Nanotechnology, Nanoparticles
and Nanononsense

Mark Bumiller
Definitions*

- **nanoparticle, n**—*in nanotechnology*, a sub-classification of ultrafine particle with lengths in two or three dimensions greater than 0.001 micrometer (1 nanometer) and smaller than about 0.1 micrometer (100 nanometers) and which may or may not exhibit a size-related intensive property.

- **fine particle, n**—*in nanotechnology*, a particle smaller than about 2.5 micrometers and larger than about 0.1 micrometers in size.

- **ultrafine particle, n**—*in nanotechnology*, a particle ranging in size from approximately 0.1 micrometer (100 nanometers) to .001 micrometers (1 nanometer).

*ASTM E 2456-06 Standard Terminology Relating to Nanotechnology*
The Lycurgus Cup - Late Roman, 4th century AD
The Lycurgus Cup - Late Roman, 4th century AD
Gold Nanoparticles

Gustav Mie, Ann. Physik 25, 377 (1908)

Surface Plasmon Resonance
Gold Nanoparticles In Use

- Pregnant women have excess of hormone HcG*
- HcG binds to complementary DNA base pair sequence
- That lock for HcG key is attached to gold nanoparticles
- Those gold nanoparticles reflect light of specific color
- If HcG detected: line reflects red

*human gonadotropin hormone
Foreseen in 1959 by Richard Feynman

The first use of the concepts in 'nano-technology' (but predating use of that name) was in "There's Plenty of Room at the Bottom," a talk given by physicist Richard Feynman at an American Physical Society meeting at Caltech on December 29, 1959.

But I am not afraid to consider the final question as to whether, ultimately---in the great future---we can arrange the atoms the way we want; the very atoms, all the way down! What would happen if we could arrange the atoms one by one the way we want them
First Used in 1974 Paper in Japan

The term "nanotechnology" was defined by Tokyo Science University Professor Norio Taniguchi in a 1974 paper*:

“Nano-technology” mainly consists of the processing of, separation, consolidation, and deformation of materials by one atom or by one molecule.

What is Nanotechnology?

So what is Nanotechnology?*

Nanotechnology is the understanding and control of matter at dimensions between approximately 1 and 100 nanometers, where unique phenomena enable novel applications. Encompassing nanoscale science, engineering, and technology, nanotechnology involves imaging, measuring, modeling, and manipulating matter at this length scale.

Where does HORIBA fit in?

National Nanotechnology Initiative (NNI)

- Established in 2001
- Framework for a comprehensive nanotechnology R&D program
- Foster transfer of new technologies into products
- Develop & sustain educational resources, a skilled workforce, and the supporting infrastructure and tools to advance nanotechnology.

Table 1 NNI Budget, 2007 - 2009 (dollars in millions)

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<th>2007 Actual</th>
<th>2008 Estimate*</th>
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<td><strong>TOTAL</strong></td>
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<td>1,491</td>
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</table>
NNI Research Centers
Scale

Carbon nanotube D=1nm

Human hair D=100 μm
= 100,000 nm

X 100,000

1 nm

100 μm
What is Nanotechnology?

- **Dry**: Fabrication of structures in carbon, silicon, other inorganics (nanoparticles)
- **Wet**: Biological systems in a water environment. Genes, proteins, enzymes (biotechnology)
- **Computational**: modeling & simulation (university research)
Industry Focus

NanoTechnology Industry Focus

Yes

MicroElectroMechanical (MEMS) devices

No

Information Technology 20%

Semiconductor 17%

Nano MEMS 11%

Other 10%

Medical/Healthcare 8%

Hybrid Materials 34%
Table 2: Index Constituents (3/19/04)

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*Update 7/21/09
divide all values by (at least)2

LA-950 Customer: Nanophase
Companies & Stocks

Lux Nanotech Index*

The Nanotechnology Value Chain

Nanomaterials
- Nanoscale structures in unprocessed form
  - Nanoparticles, nanotubes, quantum dots, fullerenes, dendrimers, nanoporous materials, etc.

Nanointermediates
- Intermediate products with nanoscale features
  - Coatings, fabrics, memory and logic chips, contrast media, optical components, orthopedic materials, superconducting wire, etc.

Nano-enabled products
- Finished goods incorporating nanotechnology
  - Cars, clothing, airplanes, computers, consumer electronics devices, pharmaceuticals, processed food, plastic containers, appliances, etc.

Nanotools
- Capital equipment and software used to visualize, manipulate, and model matter at the nanoscale
  - Atomic force microscopes, nanoimprint lithography equipment, molecular modeling software, etc.

* From www.luxresearchinc.com
<table>
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<th>Component Name</th>
<th>Ticker</th>
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### Companies & Stocks

#### Lux Nanotech Index

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#### End-Use Incumbents

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<td>LUXIII</td>
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#### 52 Week

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<td>16 Jun 2005</td>
<td>10 Jul 2009</td>
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Making Nanoparticles

**Top Down**
- Make particles smaller

**Bottom Up**
- Build from atomic or molecular level up

Self assembly of micelles
Drug Particles

- Many new chemical entities (NCEs) are poorly water soluble.
- Advances in Drug Discovery (high throughput screening) produce 30%-50% poorly water soluble leads.
- Elan-NanoSystems™ technology reduces the particle size of a poorly water soluble drug from several microns to ~50 nm.
- Reduction in particle size results in an increase in surface area.

Assumption: 30% of discovery actives are water insoluble & not evaluated in animal studies because of formulation problems.
Dissolution & Particle Size

\[ \frac{dc}{dt} = k S \left( C_s - C_t \right) \]

dc/dt = rate of dissolution
k = dissolution rate constant
S = surface area of dissolving drug
C_s = saturation concentration of drug in diffusion layer
C_t = concentration of drug in dissolution medium @ time t

To increase dissolution rate:
S may be easiest parameter to adjust
Decrease particle size, increases surface area
Oral Absorption of Poorly Soluble Drugs

Dissolution time $\gg$ GI transit time

Dissolution time $<\ GI$ transit time
Blood Absorption Profile

- NanoCrystal® Colloidal Dispersion
- NanoCrystal® Solid Dosage Form
- Commercial Tablet

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<th>Product/Licensee</th>
<th>Technology</th>
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<td>Elan Drug Technologies</td>
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Top Down: Elan NanoMill
Top Down: Elan NanoMill
Size Reduction Measured on LA-950

NanoMill-10 Particle Size vs. Mill Residence Time

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<th>Size (nm)</th>
<th>Time (min)</th>
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<td>5,240</td>
<td>0.82</td>
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<tr>
<td>406</td>
<td>5.6</td>
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<td>240</td>
<td>18.1</td>
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<td>155</td>
<td>26.3</td>
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Frequency (%) vs. Particle Size (microns)
Top Down: Microfluidizer*

- Continuous processing
- Accommodates materials with high solid content
- Certain models accommodate two separate stream feeds
- High pressure used to deliver product into the interaction chamber
- Constant pressure pumping system
- Microchannels range from 50-500 microns
- Unsurpassed shear and impact forces
- Temperature regulated by a heat exchanger
- Lack of moving parts maximizes uptime
- Repeatable and scalable results

* See http://www.microfluidicscorp.com/
Top Down: Microfluidizer
Particle size reduction – Colitis drug nanoemulsion

Median particle size (D50) after processing: 385 nm
Median particle size (D50) after 1 pass @ 24,000 psi: **96 nm**
Bottom Up: Microfluidics Reaction Technology (MRT)

Novel combination of bottom-up process development and an impinging jet processor achieves unparalleled and unprecedented results

Applications of MRT
• Crystallization
• Nanoencapsulation
• Chemical reactions
• Process intensification

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<tr>
<th>API</th>
<th>MRT Bottom-up Crystallization 1 Pass (nm)</th>
<th>Beaker Bottom-up Crystallization (nm)</th>
<th>Top-down Particle Size Reduction 25 Passes (nm)</th>
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<td>180</td>
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<td>Oxcarbazepine</td>
<td>767</td>
<td>5,000x20,000</td>
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<td>Loratadine</td>
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Report: Azithromycin
Z-average particle size: 82 nm
LA-950
Low End Sensitivity

30, 40, 50, 70 nm latex standards
Low End Sensitivity

Sensitivity: small particle detection

30 nm silica

- S.P.Area: 2.0183E+6 cm²/cm³
- Mean Size: 0.02990 μm
- Variance: 5.0313E-6 cm²
- Median Size: 0.03013 μm
- Mode Size: 0.0302 μm
- Skewness: -0.2901

40 nm latex

- S.P.Area: 1.4253E+6 cm²/cm³
- Mean Size: 0.04241 μm
- Variance: 1.2759E-5 cm²
- Median Size: 0.04214 μm
- Mode Size: 0.0422 μm
- Skewness: -0.1514
Nanoparticles for Drug Delivery

“Engineered Particles”
Bind Biosciences*

Targeting ligand provides recognition, enabling targeted nanoparticles to identify and bind to their intended target site.

Surface functionalization shields targeted nanoparticles from the immune system.

Polymer matrix encapsulates payload molecules in a matrix of biodegradable polymers.

Therapeutic payloads include small molecules, peptides, proteins, etc.

* Cambridge, MA, recent LA-950 customer
Bob Langer (MIT Professor)

- 600 patents, sublicensed to over 200 pharmaceutical/biotech companies, almost all particle technology related
- Founded over 24 companies (so far)
- 160 major awards: National Medal of Science, National Inventors Hall of Fame, National Academy of Engineering and the National Academy of Sciences (at age 43)
Quantum Dots

2nm — 6 nm

5-75 atoms in diameter

Explore the future
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Quantum Dots Applications
Self Assembly: Micelles

- Hydrophobic tail
  - non polar
  - \(-\text{c-h-c-h-c-h-}\)
  - hates water

- Hydrophilic head
  - polar
  - loves water

Aqueous solution

Organic solvent

Explore the future
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Liposomes

Bilayer sheet

Micelle

Liposome

Hydrophilic head

Aqueous solution

Hydrophobic tail

100 nm
**DLS Application: Liposomes**

- Liposome particle size after 5 passes through a 100 mm membrane ~ 250 nm
- Liposome particle size after 20 passes through a 100 mm membrane ~ 150 nm

**LB-550 DLS System**

*Image of a liposome structure with labels for polar ends, non-polar bilayer, and hydrophilic pocket.*
FDA Approved Liposome: Doxil
FDA Approved Nanoparticle: Abraxane

- Nanoparticle formulation of the chemotherapy drug paclitaxel and the protein albumin
- More effective and less toxic than the free form of the drug
Fullerenes

$C_{60}$: Buckyballs

Cylindrical fullerene: carbon nanotube
Carbon Nanotubes

Discovered by:
Sumio Iijima, 1991

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<td>~1 (from 1 to 5)</td>
<td>13-53(^E)</td>
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<td>Armchair SWNT</td>
<td>0.94(^T)</td>
<td>126.2(^T)</td>
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<td>Zigzag SWNT</td>
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<td>Chiral SWNT</td>
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<td>MWNT</td>
<td>0.8-0.9(^E)</td>
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<td>Stainless Steel</td>
<td>~0.2</td>
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<td>Kevlar</td>
<td>~0.15</td>
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<td>Kevlar(^T)</td>
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Carbon Nanotube Applications

- **Structural**
  - Bulletproof vests, waterproof & tear resistant clothes, concrete, space elevator

- **Electromagnetic & circuits**
  - Nanowire, transistors, superconductors

- **Chemical**
  - Filters, hydrogen storage
Particle Stability

- Stable suspension
- Sedimentation
- Flocculation
- Stable emulsion
- Coalescence
- Creaming

Diagram showing negatively charged particle surface, zeta potential, and slipping plane.
Particle Stability: DT-1200
The nanofiller particles in Adper™ Single Bond Plus Adhesive are added in a manner that does not allow them to cluster together. The particles are stable and will not settle out of dispersion. So unlike some filled adhesives, Adper Single Bond Plus adhesive does not require shaking prior to use.

Nanoparticle Safety: Cosmetics

- “Preliminary scientific research has shown that many types of nanoparticles can be toxic to human tissue and cell cultures…”
- Suggests banning all cosmetics with particles <100nm
- Many sunscreens contain ZnO particles <100 nm
Nanoparticle Safety

- NGO Friends of the Earth issue report requesting ban on all skin care products w/particles <100nm
- Claim: some nano-articles are dangerous, so ban all
- Industry claims some nano-particles are safe, so don’t worry
- FDA says we only believe data in peer reviewed journals, but form task force to investigate
LA-950 Data: % Below 100 nm

- LA-950 can measure down to nanoparticle range (<100 nm)
- Plus up to mm range
- Ideal instrument to detect nanoparticles in presence of larger particles
No Dilution: Acoustic Spectroscopy

![Graphs showing the comparison of attenuation and PSD for zinc oxide A in solvent and zinc oxide B in water.](image-url)
No Dilution: Acoustic Spectroscopy

Diameter [um]

PSD, weight basis

- Zinc oxide, reagent ACS by Acros Organics
- Zinc oxide, 99.5+% Acros Organics
- Z50-500 USP
- Z52-500 USP
- S80249 by Fisher Scientific
- Zinc oxide 99.99% by Alfa Aesar
- Zinc oxide ACS MO Biomedicals, LLC
- PolystormorTM by Mallinckrodt Chemicals
- Nanopowder America Elements
No Dilution: Acoustic Spectroscopy

Percentage of nano particles in Z52-500 sample after incremental additions of the Mallinckrodt Chemicals sample. The X-axis is a percentage calculated from the known amount of the added Mallinckrodt sample, assuming that it contains 20% on nano-particles, according to the Table 1. The Y-axis is a percentage calculated from the attenuation spectra, which is measured for the mixture.
Nanoparticle or Not?

Details:
Silver Powder, 99.9% (metal basis)
APS: 30-50 nm
SSA: 5-10 m2/g
Particle Morphology: spherical
Crystallographic Structure: cubic

Certificate of Analysis
Silver (Ag)
APS 30-50 nm, 99.9+%  
Stock #: 0477YD

<table>
<thead>
<tr>
<th>Components</th>
<th>Contents (ppm)</th>
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<tbody>
<tr>
<td>Al</td>
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<tr>
<td>As</td>
<td>&lt; 0.5</td>
</tr>
<tr>
<td>Bi</td>
<td>20</td>
</tr>
<tr>
<td>Cu</td>
<td>17</td>
</tr>
<tr>
<td>Fe</td>
<td>56</td>
</tr>
<tr>
<td>Ni</td>
<td>3</td>
</tr>
<tr>
<td>Pb</td>
<td>9</td>
</tr>
<tr>
<td>Sb</td>
<td>&lt; 0.5</td>
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<tr>
<td>Si</td>
<td>41</td>
</tr>
</tbody>
</table>

Analytical Technique: inductively coupled plasma
Nanoparticle or Not?

SSA = $6/\rho D$

D from SEM ~50 nm
D from SSA ~60-70 nm
D from DLS ~250 nm
So: is this a nanoparticle?

Used ultrasound to disperse to primary particles or use weak acid to break bonds
D from DLS ~50 nm
Nanoparticle or Not?
Volume vs. Number Distribution
Nanoparticle or Not?
Sample Prep Matters: Ultrasound

![Diameter vs. % by Volume Graph](image)

<table>
<thead>
<tr>
<th>Data Name</th>
<th>Graph Type</th>
<th>Median Size (µm)</th>
<th>Mean Size (µm)</th>
<th>Std. Dev. (µm)</th>
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<tbody>
<tr>
<td>mineral oil as is-1</td>
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<td>0.16866</td>
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</table>

Explore the future
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Conclusions

- Many techniques can measure <100 nm
  - DLS when all sample <1 μm
    - LB-550
  - Diffraction for below + above
    - LA-950
  - Acoustic spectroscopy for undiluted tests
    - DT-1201

- Beware that choice of techniques, sample prep & report basis influence results
Nanononsense

Grey goo is a hypothetical end-of-the-world scenario involving molecular nanotechnology in which out-of-control self-replicating robots consume all matter on Earth while building more of themselves—a scenario known as ecophagy.

Remember Ice-9?