Current Research and Development of Nanometrology in Thailand

“Experience and Practices in the Testing, Characterization, Standardization and Certification of Nanoproducts”

Annop Klamchuen

Head of Nano Characterization Laboratory (NCL) National Nanotechnology Center (NANOTEC), Thailand
Outlines

1. Current Status of Nano Products
2. Nano Products Characterization
   - Guidelines & Best Practices from THAILAND –
3. Important of Traceability
   - Preliminary Work on Inter-Laboratory Comparison-
4. Certification
   - Best practices and experiences from THAILAND -
CURRENT STATUS OF

Nano Products
Many nano products are being developed and marketed without detailed characterization nor prior review and approval of their efficacy and safety.
Characterization & Regulatory Gaps of Nano Products

• No agreed protocols for physico-chemical characterization

• Existing ‘methods of test’ may not be suitable for nanoscale devices and dimensions

• Measurement techniques and instruments need to be developed and/or standardized

• Calibration procedures and CRMs needed for validation of test instruments at nanoscale
Nanotechnology may become a new non-tariff barrier
LACK OF INFORMATION

INFORMATION OF THE TEST ITEM (CLOSED TO) CORRECT ONES

• WHICH PART IS CLAIMED NANO?
• COMPOSITION OF THE NANO
• FUNCTION CLAIMED

REAL or FAKE?
REAL with QUALITY?
REAL with SAFETY?
WHERE IS MY NANOMATERIALS?

Figure 6. The categorization framework for nanomaterials. The nanomaterials are categorized according to the location of the nanostructure in the material (Hansen et al. 2007).
NATIVE STATE >> COMPLEX MATRIX

a

SPF 30+

? 

b

?
NATIVE STATE >> COMPLEX MATRIX

PURE NANOMATERIALS

Sample 15
Sample 28
Sample 32
Sample 55
WHICH INSTRUMENT BEST?
<table>
<thead>
<tr>
<th>Test Method</th>
<th>Titles</th>
</tr>
</thead>
<tbody>
<tr>
<td>ISO 22916: 2011</td>
<td>Antimicrobial products – Test for Antimicrobial Activity and Efficacy</td>
</tr>
<tr>
<td>JIS Z 2801: 2006</td>
<td>Test for Antimicrobial Activity of Plastics</td>
</tr>
<tr>
<td>AATCC 100 (2004)</td>
<td>Assessment of Antimicrobial Finishes on Textile Materials</td>
</tr>
<tr>
<td>ASTM E2149-10</td>
<td>Determining the Antimicrobial Activity of Immobilized Antimicrobial Agents under Dynamic Contact Conditions</td>
</tr>
<tr>
<td>AATCC 147 (2011)</td>
<td>Antibacterial Activity Assessment of Textile Materials: Parallel Streak Method</td>
</tr>
<tr>
<td>JIS L 1902: 2008</td>
<td>Testing for Antibacterial Activity and Efficacy on Textile Products</td>
</tr>
<tr>
<td>NCCLS M2-A6 (DISK)</td>
<td>Antimicrobial Disk Susceptibility Tests</td>
</tr>
</tbody>
</table>
NanoProducts
CHARACTERIZATION
- Guidelines & Best Practices from THAILAND -
National Advanced Nano-characterization Center (NANC)
OUR FACILITIES

Nano Imaging Laboratory
Advanced Optical Microscopy & Spectroscopy Laboratory
Nanoparticle Detection & Sizing Laboratory
Integrated NanoMaterials Characterization Laboratory
Analytical NanoChemistry Laboratory
NanoProduct Functionality & Specification Laboratory
Nanoproduct safety Laboratory
Flagship: NanoMARKS

“Nanometrology” & “Nanosafety”

Co-R&D
Nano-characterization Laboratory (NCL)
Nano Safety and Risk Assessment Laboratory (SRA)

NANC
National Institute of Metrology Thailand

“Micrometrology” & “Chemical safety”

Protocol
- Stakeholder panel
- Sign an “NDA”
- Representative from NANOTEC
- Workshop and training

Standard
- Reference Materials
- Inter-lab comparison
- Workshop and Training
Testing of Products Containing Nanomaterials

Thailand is presently establishing of standard testing and characterization services to specific requirements of targeted industrial sectors.

<table>
<thead>
<tr>
<th>Food</th>
<th>Cosmetic</th>
<th>Petrochem.</th>
<th>Textile</th>
</tr>
</thead>
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<tr>
<td><img src="image" alt="Food" /></td>
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<td><img src="image" alt="Textile" /></td>
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</tbody>
</table>

**Assay Cascade Protocols**

1. Physico-chemical characterization of nanomaterials
2. Functionality / Performance of nanoproducs
3. Safety assessment of nanomaterials

**Test Parameters**

- Size
- Shape
- Surface area
- Agglomeration state
- Structure
- Composition
- Surface chemistry
- Surface activity
- Crystal structure
- Solubility
- Antibacterial activity
- Water repellent activity
- Etc.
- Skin irritation/ corrosion
- Cytotoxicity
- Phototoxicity
- Genotoxicity
- Ecotoxicity
### CASCADE ANALYSIS OF NANOPRODUCT

<table>
<thead>
<tr>
<th>Sectors</th>
<th>Assay Cascade Protocols</th>
</tr>
</thead>
</table>
| **Foods/ Food Additives / Agricultural products** | - Physical-chemical characterization of nanomaterials  
- Functionality / Performance of nanoproducts  
- Release test  
- Biological and cell-based model assays  
- Cytotoxicity  
- Absorption  
- Metabolism: interaction with hepatic enzyme  
- Immunotoxicity  
- Genotoxicity (Comet assay and micronucleus assay) |
| **Paints / Coating** | - Physical-chemical characterization of nanomaterials  
- Functionality / Performance of nanoproducts  
- Release test  
- Health: Cell-based model assays  
- Penetration test  
- Acute toxicity assay  
- Immunotoxicity assay  
- Genotoxicity (Comet assay and micronucleus assay)  
- Environment: Ecotoxicity |
## CASCADE ANALYSIS OF NANOPRODUCT

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</table>
| Cosmetics/ Herbal products / Medical Devices | • Physical-chemical characterization of nanomaterials  
• Functionality / Performance of nanoproducts  
• Biological and cell-based model assays  
• Skin penetration test *(in vitro)*  
• Acute skin irritation test *(in vitro)*  
• Cytotoxicity *(in vitro)*  
• ROS assay *(in vitro)*  
• Immunotoxicity *(in vitro)*  
• Genotoxicity (Comet assay and micronucleus assay) |
| Textiles & Clothing              | • Physical-chemical characterization of nanomaterials  
• Functionality / Performance of nanoproducts  
• Release test  
• Biological and cell-based model assays  
• Acute skin irritation test *(in vitro)*  
• Cytotoxicity *(in vitro)*  
• ROS assay *(in vitro)*  
• Immunotoxicity *(in vitro)*  
• Genotoxicity (Comet assay and micronucleus assay) |
### CASCADE ANALYSIS OF NANOPRODUCT

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<tr>
<td><strong>Petrochemicals</strong></td>
<td>- Physical-chemical characterization of nanomaterials</td>
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</table>

| **Computer hardware & Electronic devices** | - Physical-chemical characterization of nanomaterials                                   |
|                                           | - Functionality / Performance of nanoproducts                                           |
|                                           | - Micro/ Nanostructure                                                                  |
|                                           | - Surface chemistry,                                                                    |
|                                           | - Chemical composition                                                                 |
|                                           | - Contamination                                                                       |
|                                           | - Failure analysis                                                                     |

*Solder balls jetted onto suspension pads with poor wettability*
Effect of physicochemical characteristics of nanomaterials on their toxicities

**5S Factor**

- Size
- Shape
- Surface area
- Surface chemistry
- Solubility
Important of Traceability
- Preliminary Work on Inter-Laboratory Comparison -
NANOTECHNOLOGY

One Measurement Accepted Everywhere
Nanoparticle Characterization - Supplementary Comparison on Nanoparticle Size

Organizer: Asia Pacific Metrology Programme (APMP)  NIMT

Reference Materials:
Gold Nanoparticle, Silver Nanoparticle, Polystyrene nanoparticles

Specific Measurements Instructions for DLS, TEM, SEM and AFM
Comparison Results

Inter-laboratory comparison on Nanoparticle Size Measurement (APMP.L-S5)
Organizer: Asia Pacific Metrology Programme (APMP)

REFERENCE MATERIALS:
GOLD NANOPARTICLE
SILVER NANOPARTICLE
POLYSTYRENE NANOPARTICLES

Specific Measurements Instructions for DLS, SEM and AFM

Measurement results of ~10 nm Gold
Measurement results of ~30 nm Silver
Measurement results of ~30 nm PLS
Measurement results of ~100 nm PLS
CERTIFICATION

- Best practices and experiences from THAILAND -
 ISO/IEC 17025 Certification Categories

**Scope 1:** Particle size analysis - Dynamic light Scattering (DLS)

**Scope 2:** Plastics - Measurement of antibacterial activity on non-porous surfaces

**Scope 3:** Particle size analysis – Image analysis by Scanning Electron Microscope (SEM)
**VOLUNTARY CERTIFICATION: Nano Q**

- Nano Q is a certified quality mark for nano products which are certified by Nanotechnology Association of Thailand.

- **Motivation to Have Nano Q**
  - **Increase Public Trust:** Facilitate healthy development of nanotechnology
  - **Protect Consumer:** Avoid waste money
  - **Protect Good Companies:** Eliminate unfair competitions
  - **Facilitate Trade:** Stimulate economic growth
<table>
<thead>
<tr>
<th>Nano-enabled Products</th>
<th>Criteria</th>
<th>Specified Properties</th>
<th>Other requirements</th>
</tr>
</thead>
</table>
| **Anti-bacterial textiles** | Final products & Raw materials (size 1 - 100 nm) | AATCC 147, AATCC-100, 1. S.aureus 2. E.coli | Anti-bacteria >99.5% After 30 washed | • SDS  
• Site visiting  
• Manufacturing process  
• Quality system  
• User Instructions  
• Reliability  
• Disclaimer |
| **Anti-bacterial paints**   | Final products & Raw materials (size 1 - 100 nm) | JIS Z 2801, ISO 22196 1. S.aureus 2. E.coli | Anti-bacteria >90% |
| **Antibacterial plastic/ceramic** | Final products & Raw materials (size 1 - 100 nm) | JIS Z 2801, ISO 22196 1. S.aureus 2. E.coli | Anti-bacteria >90% |
| **Water repellent textiles** | Final products & Raw materials (size 1 - 100 nm) | AATCC-22 Spray Method | Contact angle > 100° |
| **Water repellent paints**  | Final products & Raw materials (size 1 - 100 nm) | Contact Angle Methods | Contact angle > 100° |
| **Water repellent plastic/ceramic** | Final products & Raw materials (size 1 - 100 nm) | Contact Angle Methods | Contact angle > 100° |
The aseptic ambulance of Supremeproducts Co., Ltd has received the first NanoQ label in Thailand.

Anti-bacterial performance of nano-silver clay treated a ceiling inside an ambulance.

Figure 1 Antibacterial test for Staphylococcus aureus
(A) Non-treated nano-silver clay
(B) Treated nano-silver clay
MANDATORY CERTIFICATION

STANDARDS

Thai Industrial Standards Institute
- Standardization of nanotechnology
  - Nomenclature/terminology
  - Testing, measurement and characterization procedures
  - Health / Safety / Environment
  - Material specification
  - Guidelines and good practices
- Coordination of regulatory framework for nanoproducts

CONSUMER PROTECTION (THAILAND)
- Permissible and mandatory labeling
  - Truthful and not misleading
  - Labeling must include material information, conditions of use

FOOD & DRUG ADMINISTRATION (THAILAND)
- Pre-market approval
  For products that require an FDA approval prior to introduction to the market.
- Post-market review
  For these products, market entry and distribution are at the discretion of the manufacturer and FDA monitors the behavior of these products. Regulatory action is taken if adverse events occur.
MULTI-STAKEHOLDER ENGAGEMENT

NANOMETROLOGY

TESTING LAB

CERTIFICATION / LABELING

STANDARDIZATION

REGULATION

NANOQ
Textile / Paints/Coating / Plastics etc.

interlab. comparison

REACH nanomaterials

ICCR cosmetic directive

NanoQ

NANOTECHNOLOGY CENTER OF EXCELLENCE (8 CoE)

BIPM
National Institute of Standards and Technology
U.S. Department of Commerce

NIST

APEC

NIMT

ISO

OECD

ANF

ASIA NANO FORUM

TESTING LAB

CERTIFICATION / LABELING

STANDARDIZATION

REGULATION

MULTI‐STAKEHOLDER ENGAGEMENT