Current Research and Development of Nanometrology in Thailand

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

NANOTEC
a member of NSTDA

NSTDA

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

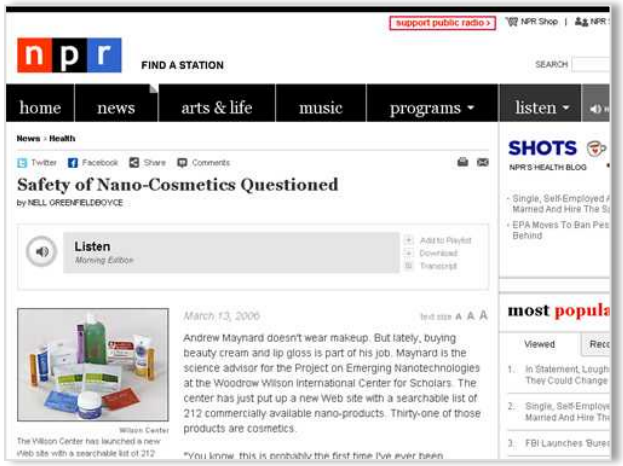
Nano Products

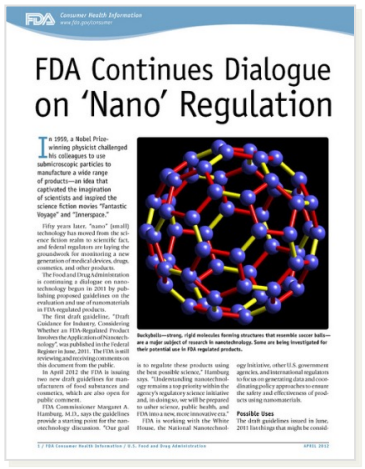
Nano Products in Market Place

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 Value Chain

Nanomaterials → **Nanointermediates** → **Nano-enable products**

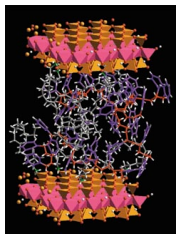
Nanoscale structures in unprocessed form

Intermediate products with nanoscale features

Finished goods incorporating nanotechnology

…






Needs !!

Test methods, Instruments, Standards, Safety

Nanotechnology may become a new non-tariff barrier

LACK OF INFORMATION

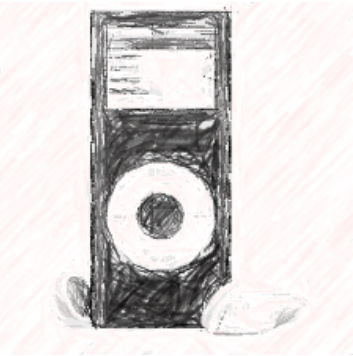
REAL or FAKE ?

REAL with QUALITY ?

REAL with SAFETY ?

INFORMATION OF THE TEST ITEM (CLOSED TO) CORRECT ONES

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



WHERE IS MY NANOMATERIALS ?

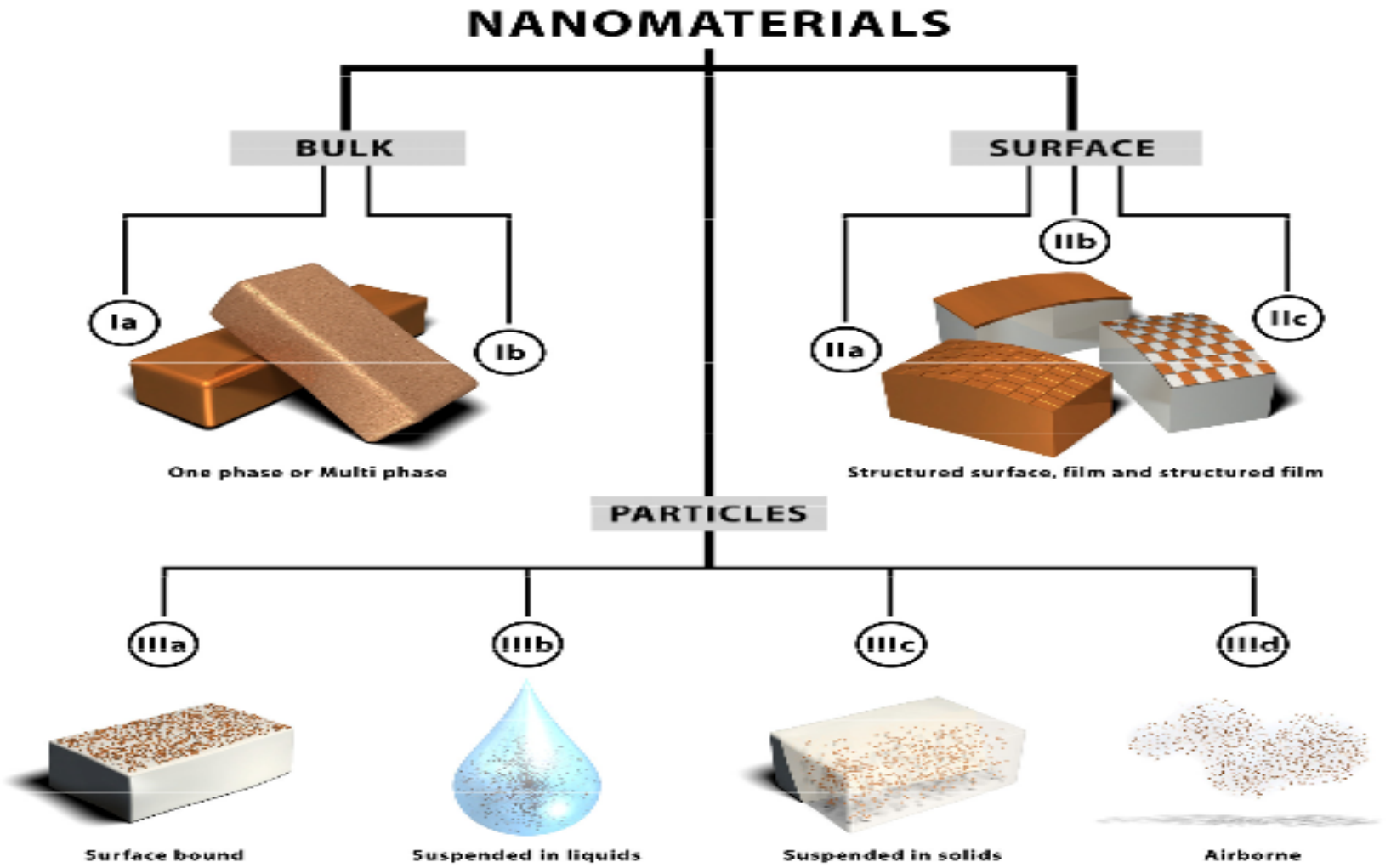
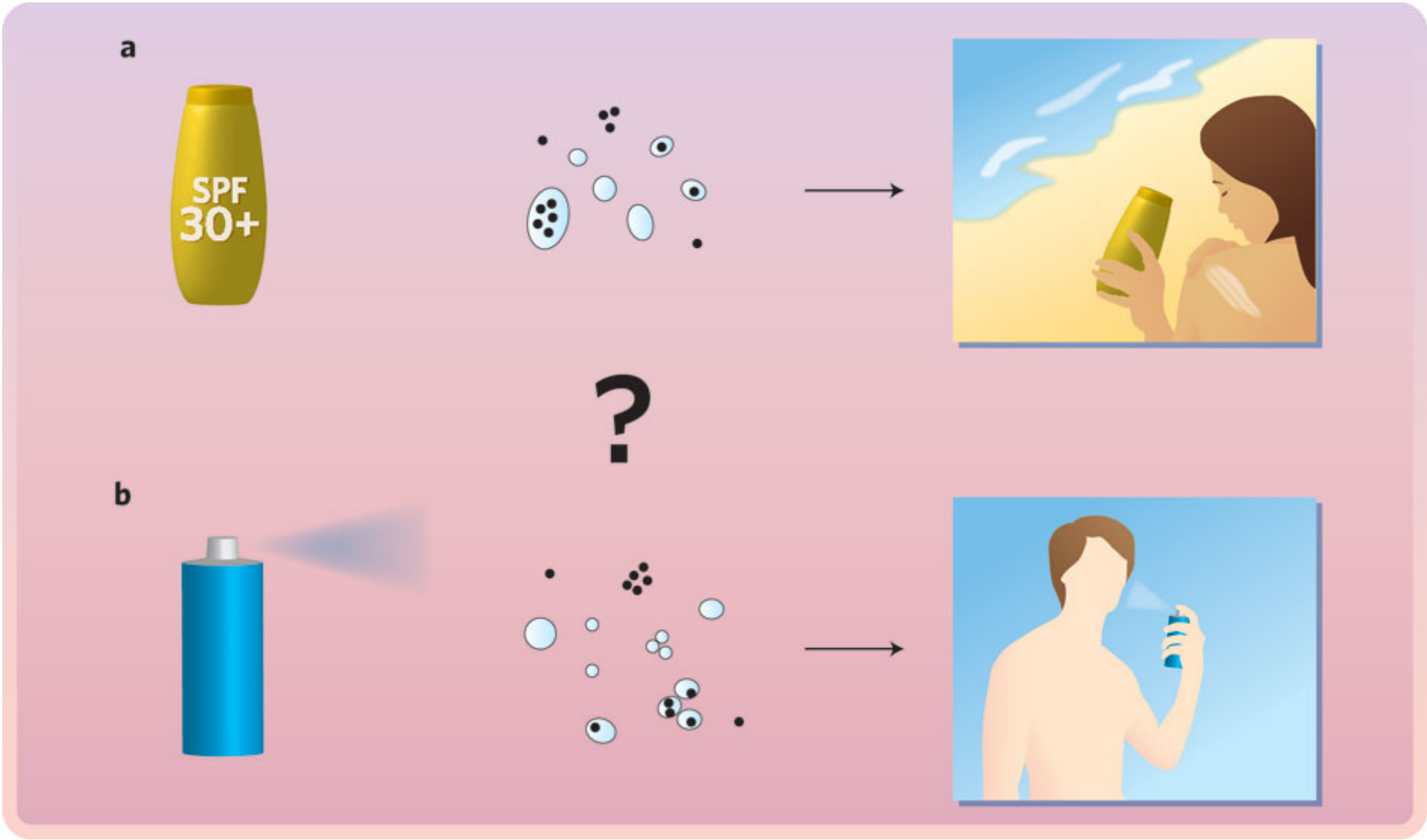
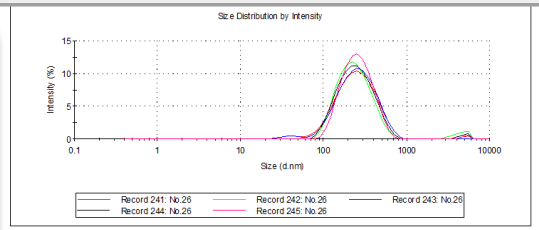


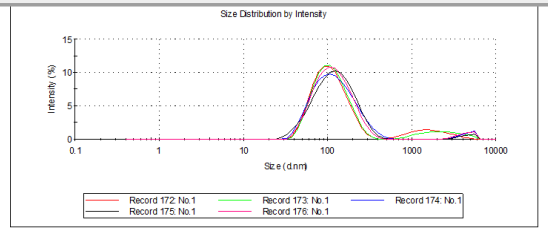
Figure 6. The categorization framework for nanomaterials. The nanomaterials are categorized according to the location of the nanostructure in the material (Hansen *et al.* 2007).

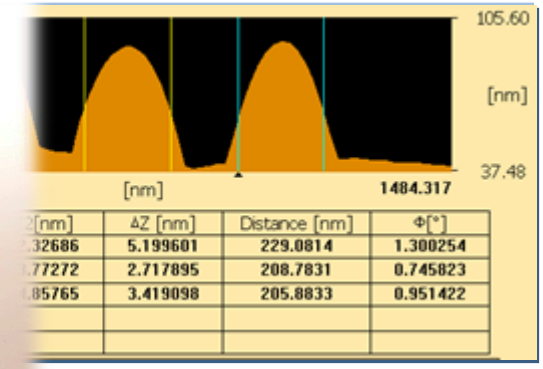
NATIVE STATE >> COMPLEX MATRIX



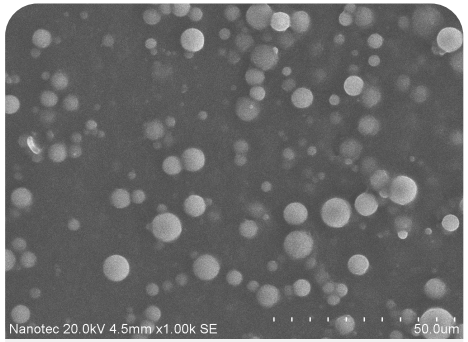
NATIVE STATE >> COMPLEX MATRIX







PURE NANOMATERIALS



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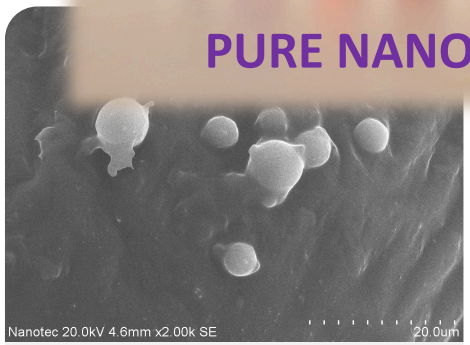

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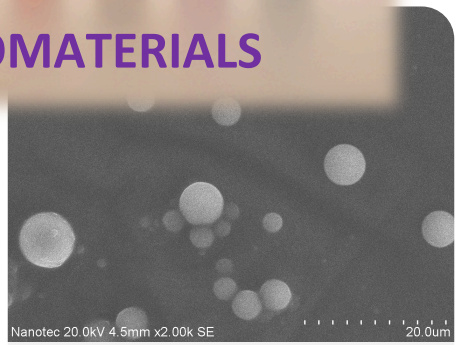
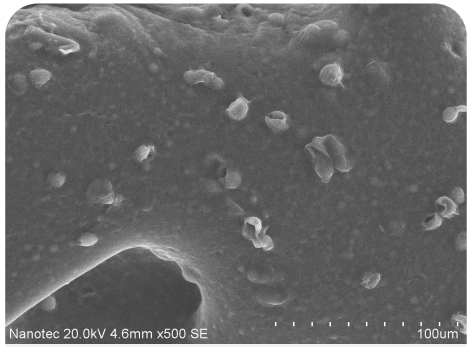
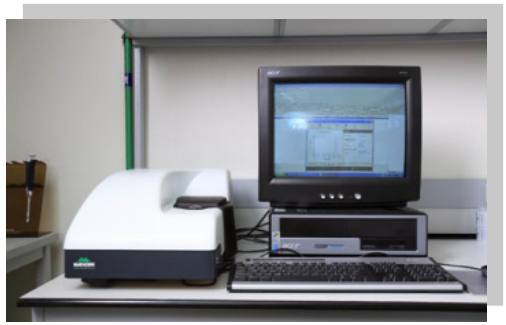
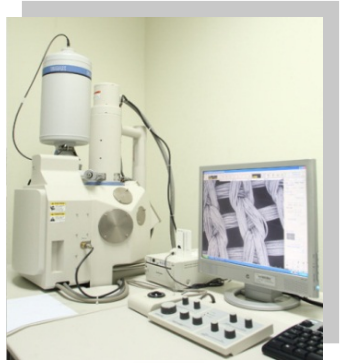
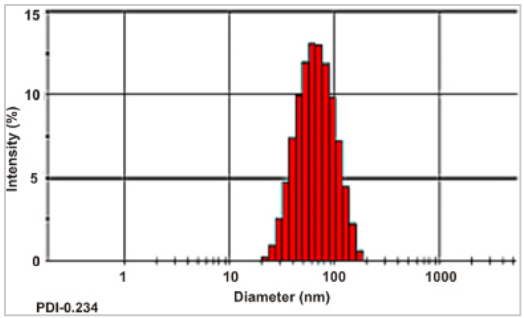
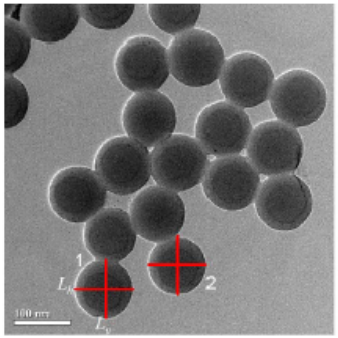
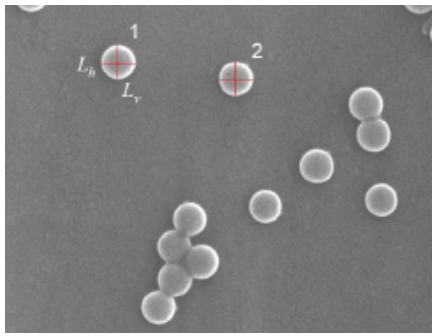
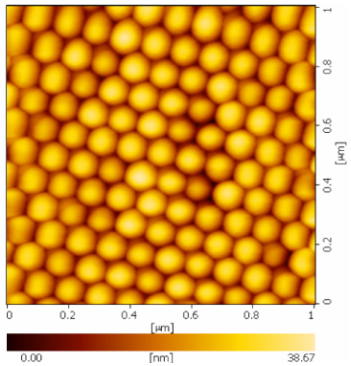


Sample 32



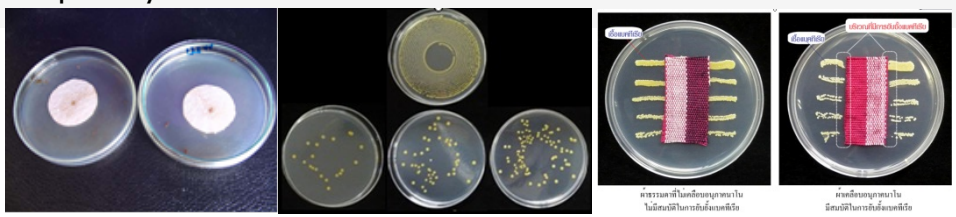
Sample 55

WHICH INSTRUMENT BEST ?

DLS	TEM	SEM	AFM
			
			

WHICH STANDARD METHOD ?

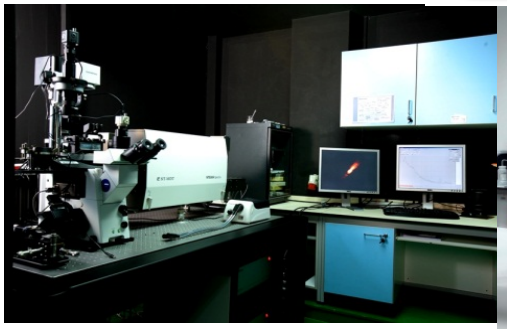
Test Method	Titles
ISO 22916: 2011	Antimicrobial products – Test for Antimicrobial Activity and Efficacy
JIS Z 2801: 2006	Test for Antimicrobial Activity of Plastics
AATCC 100 (2004)	Assessment of Antimicrobial Finishes on Textile Materials
ASTM E2149-10	Determining the Antimicrobial Activity of Immobilized Antimicrobial Agents under Dynamic Contact Conditions
CLSI M7-A7 MIC (2006)	Methods for Dilution Antimicrobial Susceptibility Tests for Bacteria that Grow Aerobically
AATCC 147 (2011)	Antibacterial Activity Assessment of Textile Materials: Parallel Streak Method
JIS L 1902: 2008	Testing for Antibacterial Activity and Efficacy on Textile Products
NCCLS M2-A6 (DISK)	Antimicrobial Disk Susceptibility Tests

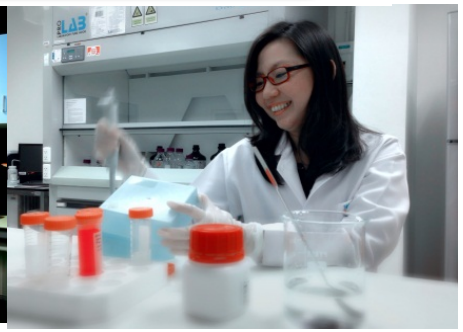


NanoProducts
CHARACTERIZATION

- Guidelines & Best Practices from THAILAND -

National Advanced Nano-characterization Center (NANC)





OUR FACILITIES

Nano Imaging Laboratory





Advanced Optical Microscopy & Spectroscopy Laboratory



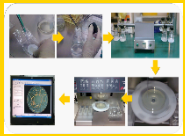
Nanoparticle Detection & Sizing Laboratory


Integrated NanoMaterials Characterization Laboratory

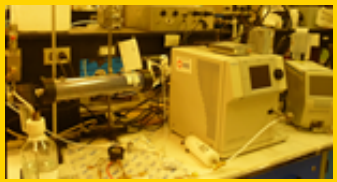


Analytical NanoChemistry Laboratory

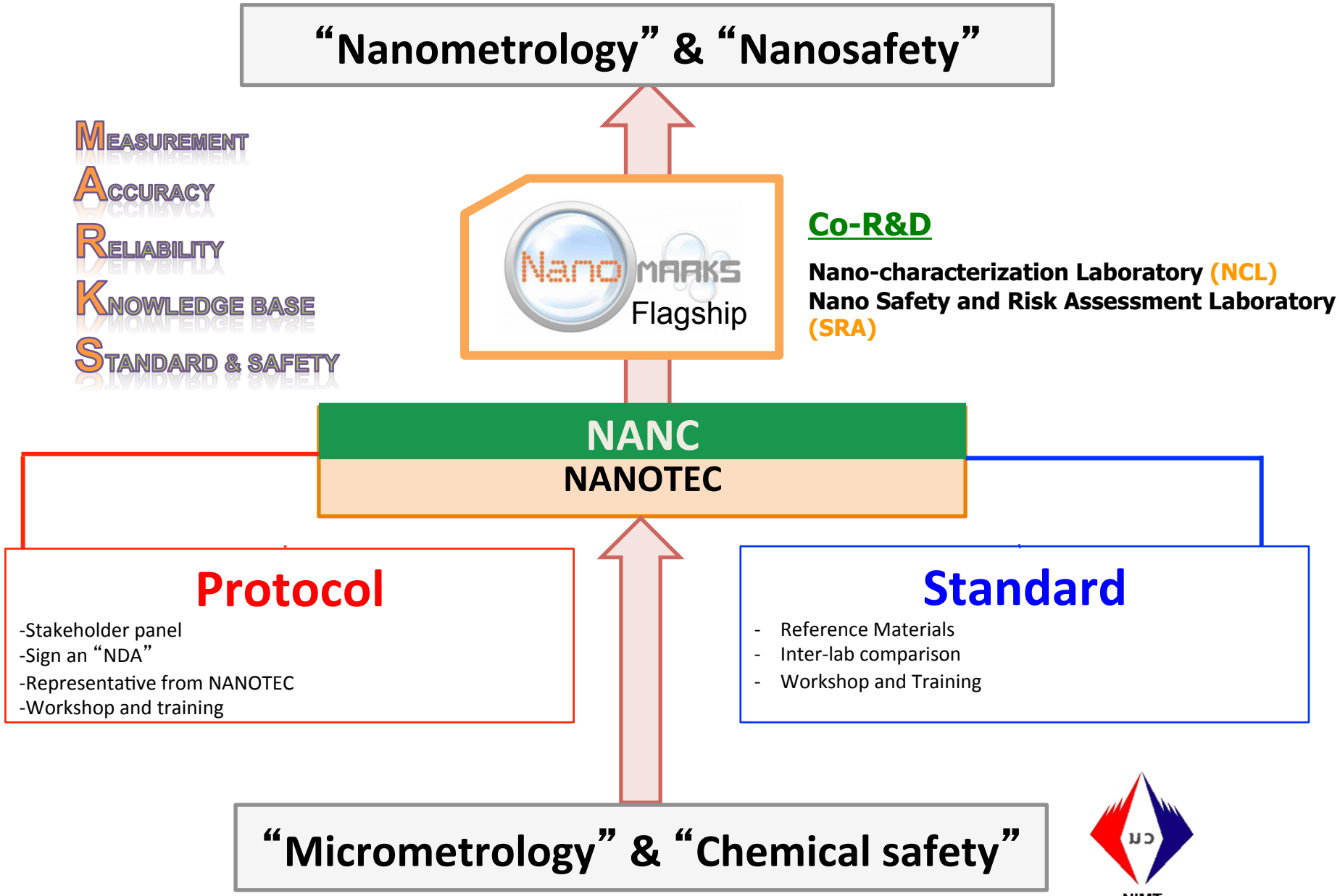

NanoProduct Functionality & Specification Laboratory



Nanoproduct safety Laboratory




Flagship: NanoMARKS



Testing of Products Containing Nanomaterials

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

Food	Cosmetic	Petrochem.	Textile
			

Assay Cascade Protocols

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

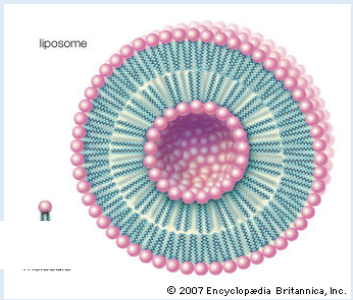
Test Parameters

❑ Size	❑ Composition	❑ Antibacterial activity	❑ Skin irritation/ corrosion
❑ Shape	❑ Surface chemistry	❑ Water repellent activity	❑ Cytotoxicity
❑ Surface area	❑ Surface activity	❑ Etc.	❑ Phototoxicity
❑ Agglomeration state	❑ Crystal structure		❑ Genotoxicity
❑ Structure	❑ Solubility		❑ Ecotoxicity

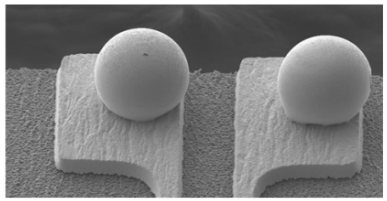
CASCADE ANALYSIS OF NANOPRODUCT

Sectors	Assay Cascade Protocols
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

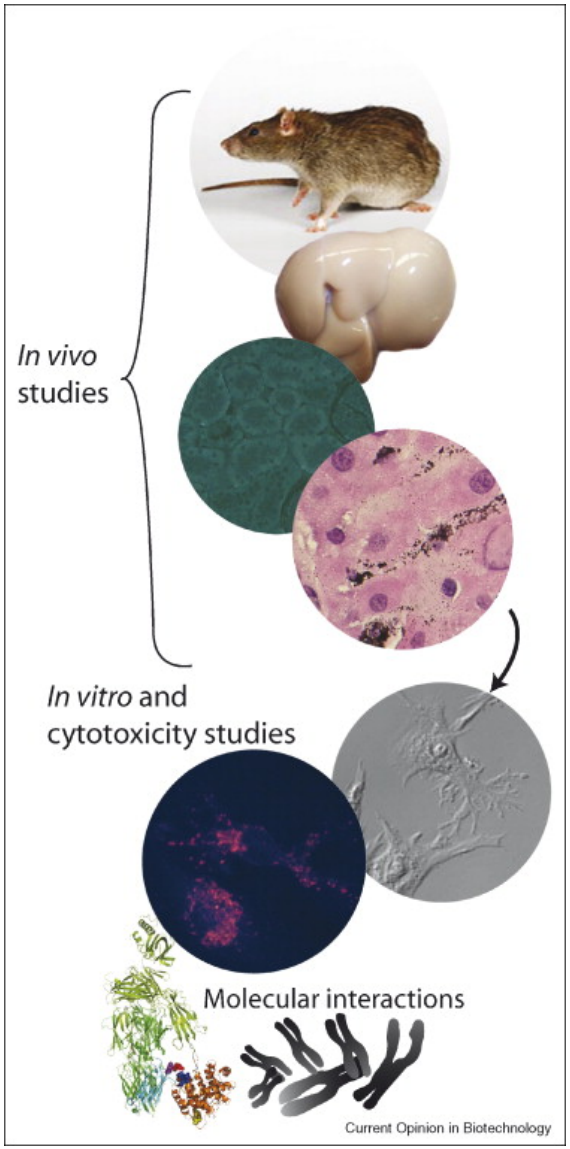
CASCADE ANALYSIS OF NANOPRODUCT

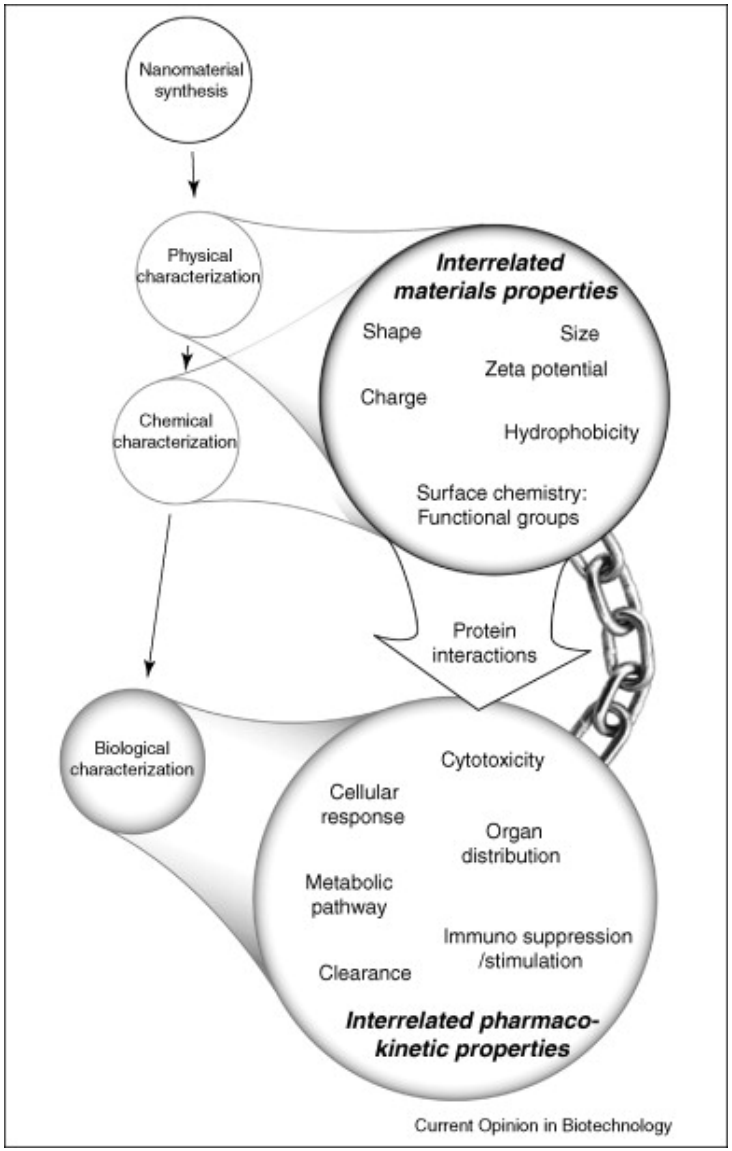
Sectors	Assay Cascade Protocols
Cosmetics/ Herbal products / Medical Devices 	· Physical-chemical characterization of nanomaterials · Functionality / Performance of nanoproducts - Biological and cell-based model assays - Skin penetration test *(in vitro)* - Acute skin irritation test (*in vitro*) - Cytotoxicity (*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

Sectors	Assay Cascade Protocols
Petrochemicals 	- 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)
Computer hardware & Electronic devices  Solder balls jetted onto suspension pads with poor wettability	- Physical-chemical characterization of nanomaterials - Functionality / Performance of nanoproducts - Micro/ Nanostructure - Surface chemistry, - Chemical composition - Contamination - Failure analysis

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

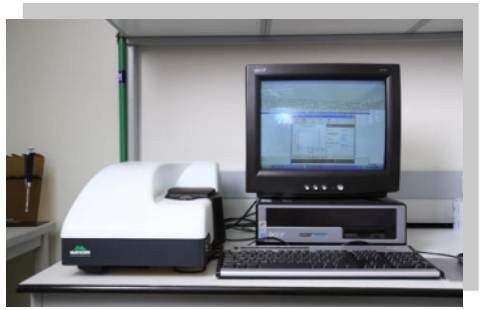
Reference Materials:

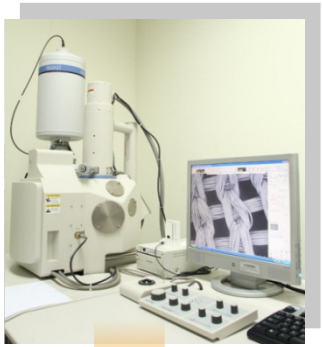
Gold Nanoparticle,
Silver Nanoparticle,
Polystyrene nanoparticles

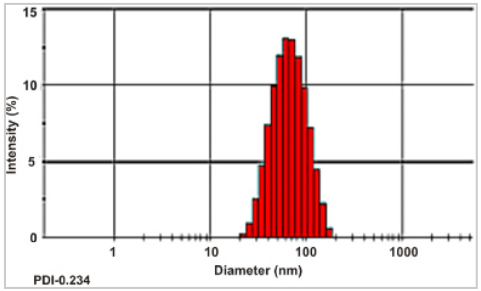
NANOTEC
a member of NSTDA

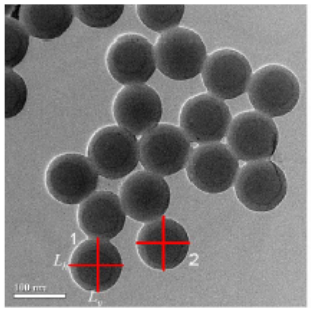
NIMT

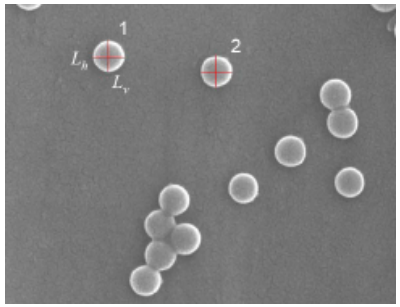
Specific Measurements Instructions for DLS, TEM, SEM and AFM

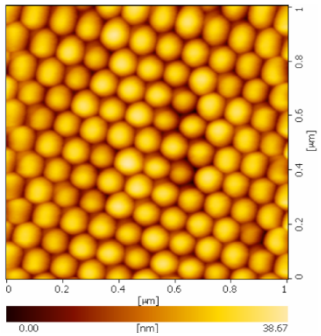












Comparison Results

Inter-laboratory comparison on Nanoparticle Size Measurement (APMP.L-S5)

Organizer: Asia Pacific Metrology Programme (APMP)

REFERENCE MATERIALS:

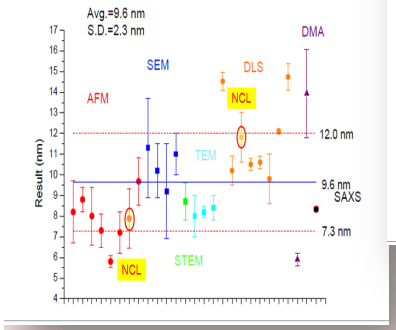
GOLD NANOPARTICLE
SILVER NANOPARTICLE
POLYSTYRENE NANOPARTICLES

NANOTEC
a member of NSTDA

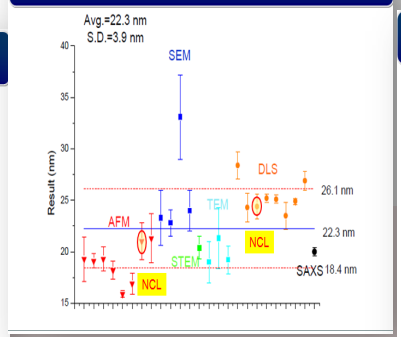
NIMT

Specific Measurements Instructions for DLS, SEM and AFM

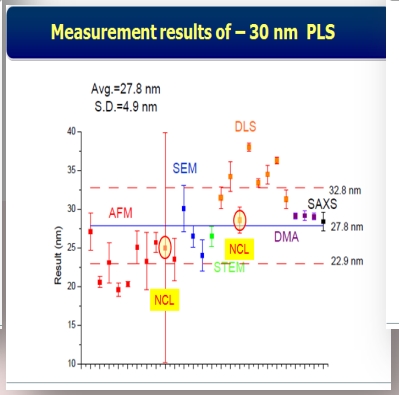
Measurement results of – 10 nm Gold



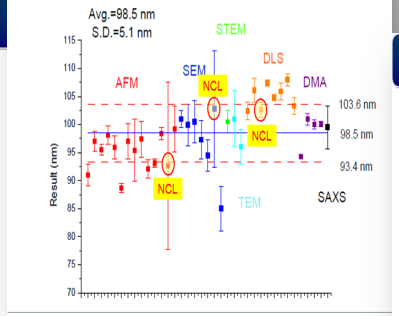
Measurement results of – 20 nm Silver



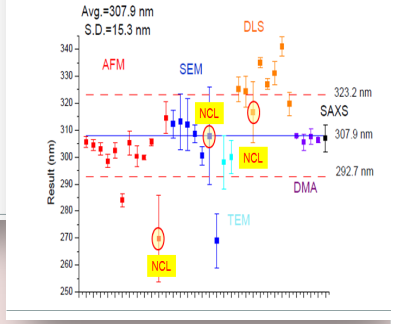
Measurement results of – 30 nm PLS



Measurement results of – 100 nm PLS



Measurement results of – 300 nm PLS

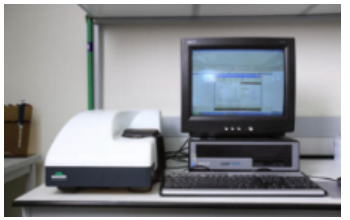


CERTIFICATION

- Best practices and experiences from THAILAND -

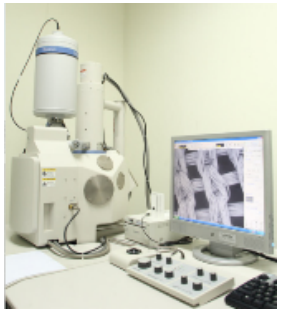
ISO/IEC 17025 Certification Categories

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



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

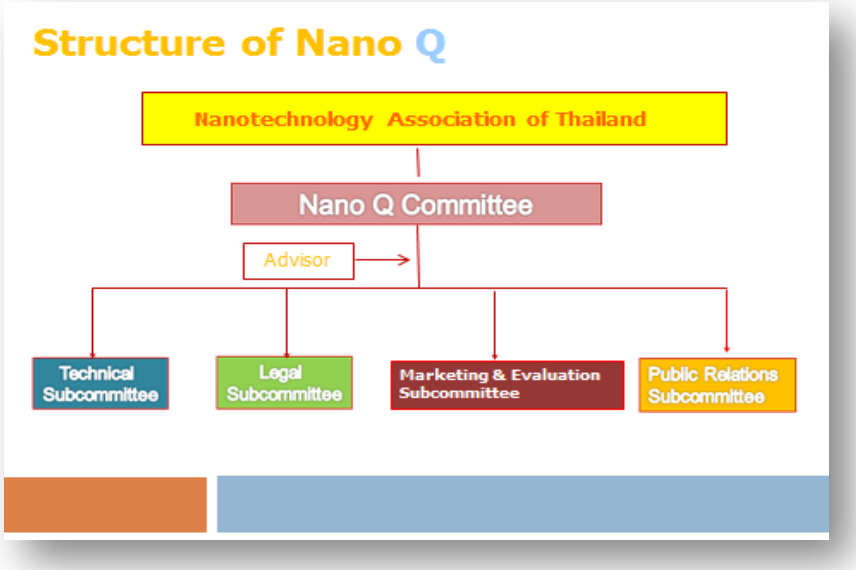
Scope 3 : Particle size analysis – Image analysis by Scanning Electron Microscope (SEM)



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**



CRITERIA FOR PRODUCT CERTIFICATION (NANO Q)

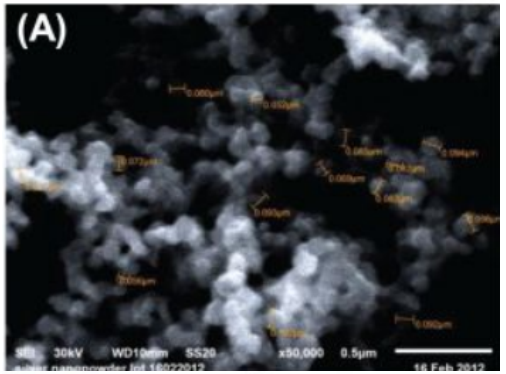
Nano-enabled Products	Criteria			
	Proof of being nanoscale material	Functional test method	Specified Properties	Other requirements
Anti-bacterial textiles	Final products & Raw materials (size 1 - 100 nm)	AATCC 147, AATCC-100, 1. *S.aureus* 2. *E.coli*	Anti-bacteria >99.5% After 30 washed	• SDS • Site 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.

supreme PRODUCTS CO.,LTD.

Sample tested for :
NANOTECHNOLOGY
✔ Anti-bacterial
Tested No. PA 55 5501
สมาคมนาโนเทคโนโลยีแห่งประเทศไทย
Nanotechnology Association of Thailand

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



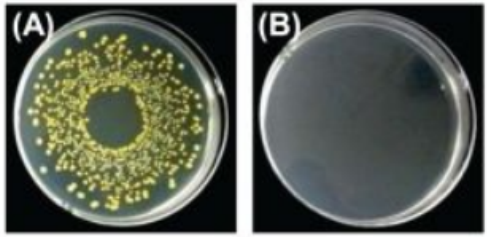


Figure 1 Antibacterial test for Staphylococcus aureus
(A) Non-treated nano-silver clay
(B) Treated nano-silver clay

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 nano-products

FOOD & DRUG ADMINISTRATION (THAILAND)

Pre-market approval

For products that require an FDA approval prior to introduction to the market.

Post-market review

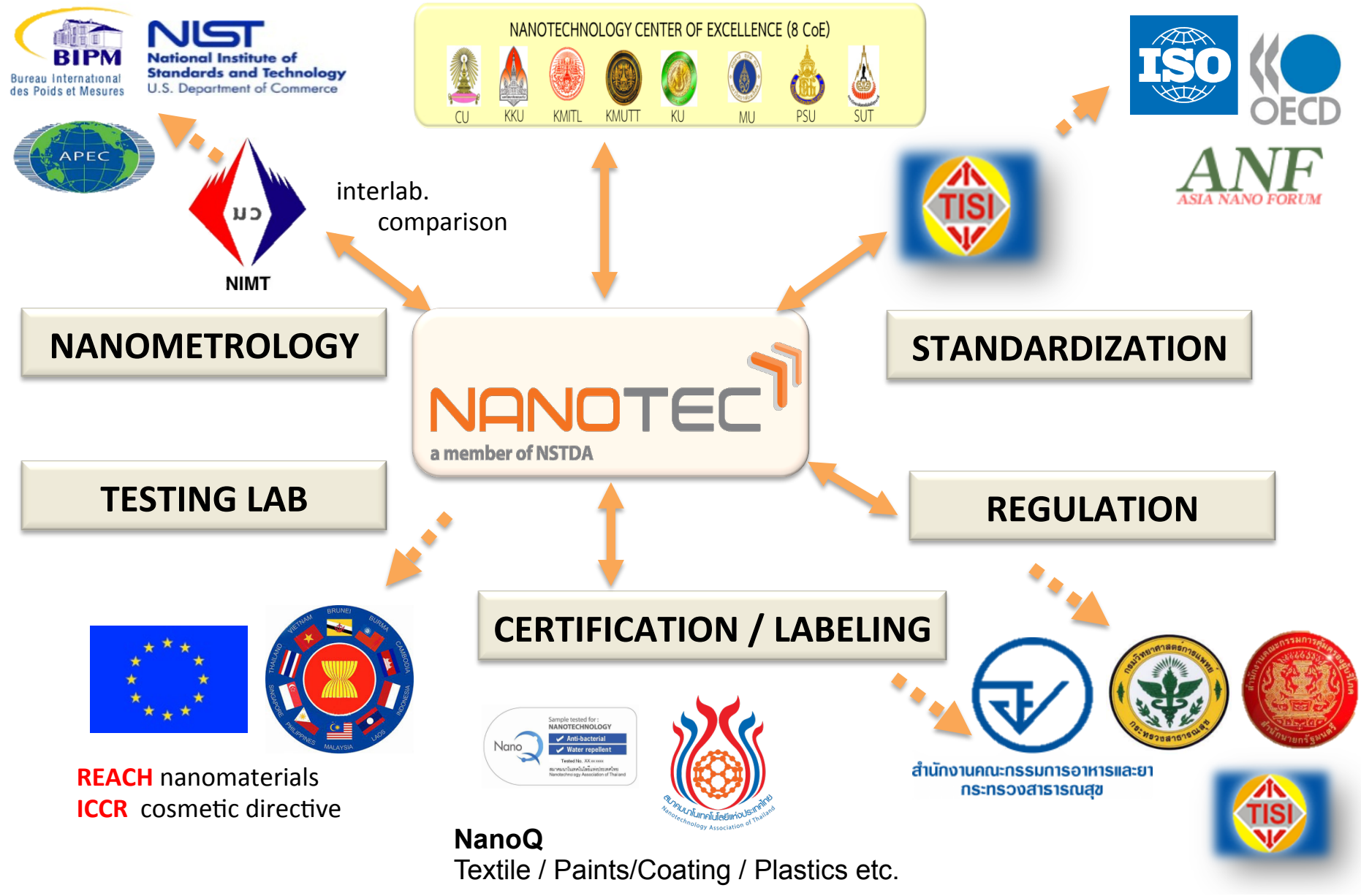
For these products, market entry and distribution are at the discretion of the manufacturer and FDA monitors the behavior of these products. Regulatory action is taken if adverse events occur.

CONSUMER PROTECTION (THAILAND)

Permissible and mandatory labeling

- Truthful and not misleading
- Labeling must include material information conditions of use

MULTI-STAKEHOLDER ENGAGEMENT



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Thank you very much for your attention