Taiwan’s NanoMark system and related activities introduction

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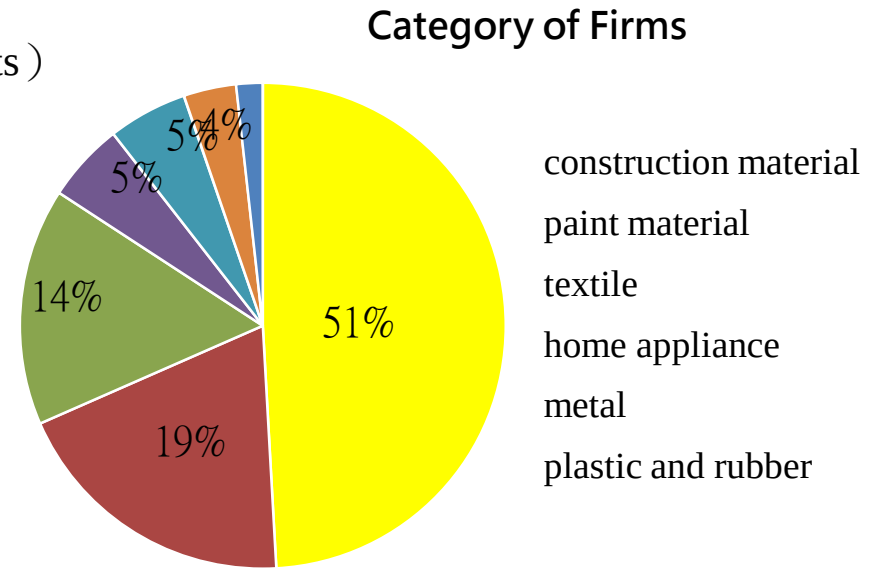
More & More Nanotech-Enabled Products Entering the Market

Examples from NanoMark application data

- **55 total certified firms（2,715 products); 44 firms are still effective (2,454 products)**
  - 30 construction material firms （2232 products）
  - 12 paint material firms（30 products）
  - 8 textile firms（27 products）
  - 3 home appliance firms（14 products）
  - 3 metal firms（10 products）
  - 2 plastic and rubber firms（132 products）
  - 1 other firms（1 products）



Need for Regulation for Nano Products !

Regulation efforts

European Union

2009 Acts: Cosmetic Regulation (EC No 1223/2009, which came into force in Jul 2013)

Regulatory Activities: Included Specific provisions for nanomaterials (definition, requirement for notification, labeling and reporting of nanomaterials).

2011 Acts: Food information to consumers regulation (EU Regulation 1169/2011), approved by the EC (July 2011) which applied from December 2014

Regulatory Activities: This regulation includes the requirement for labeling of ingredients in the form of nanomaterials (material plus word “nano” in brackets).

2012 Acts: Regulation (EC) No 1223/2009 (Article 13)

Regulatory Activities: Established the Cosmetics, Product Notification Portal (CPNP)

2013 Acts: EU Biocides Regulation

Regulatory Activities: Labeling (material plus word “nano” in brackets) and a separate evaluation of the risks deriving from nanomaterials used in various kinds of products (antifouling agents, biocides in building materials, and antimicrobial surfaces).

2015/2016 Acts: Regulation (EC) No 1223/2009 (Article 13)

EU Nano Register, REACH regulation and CLP regulation

Regulation efforts

USA

2011

Environmental Protection Agency (EPA)

Acts: Federal Insecticide, Fungicide and Rodenticide Act (FIFRA)

Regulatory Activities: EPA proposed a new approach that would consider any nanomaterial as a new active ingredient for the pesticide regulation (thus differentiating them from their macro-form).

2013

Environmental Protection Agency (EPA)

Acts: Toxic Substances Control Act (TSCA - the US regulatory provision for chemical substances

Regulatory Activities: Published a proposed Significant New Use Rules (SNUR) for 14 nanomaterials that were the subject of pre-manufacture notices (PMN). If issued in final the SNURs would require persons who intend to manufacture, import, or process new nanoscale materials based on the chemical substances designated for a Significant new use to notify EPA at least 90 days before commencing that activity and required to notify the risks

2014, 2015

Guidance documents issued

•On June 24, 2014, FDA issued three final guidance documents related to the use of nanotechnology in regulated products, including cosmetics and food substances.

•On August 5, 2015, FDA issued one final guidance document related to the use of nanotechnology in food for animals.

Regulation efforts

ASIA

Taiwan
2004
Voluntary

Iran
2009
Mandatory if apply government support

Thailand
2011
Voluntary

Malaysia
2015
Voluntary

Other Nano-Certification Programs Available Worldwide:
MNT Quality Mark, UK; Nanocertifica, Russia

Comparison between Nano-Certification Programs in Asian Countries

	NANO*Verify*, Malaysia	NanoMark, Iran	NanoMark, Taiwan	NanoQ, Thailand
1 Fee	➢ All charges borne by the company	➢ USD 300-3,000 (depending on size of the company, and analyzing cost)	➢ Varies. <USD1,600 and other cost will be bared by the government	➢ All charges beared by the company
2 Validity	➢ 2 Year	➢ 1 Year. After first supervision, increase to 3 years	➢ 3 Year	➢ 2 Years
3 Measurement Criteria	➢ Size (1-100nm)	➢ Size (1-100nm) ➢ Improvement in properties (Except for nanomaterials) ➢ Reproducibility	➢ Size (1-100nm) ➢ Unique properties	➢ Size (1-100nm) ➢ Unique nanoscale-related functionality features
4 Instruments for size characterization	➢ SEM ➢ FESEM ➢ XRD ➢ Raman ➢ TEM	➢ TEM ➢ DLS ➢ XRD ➢ FESEM / SEM ➢ ICP / AAS	➢ DLS ➢ SEM ➢ FESEM ➢ XRD ➢ Raman ➢ TEM	➢ DLS ➢ SEM ➢ More...various techniques
5 Standards used	➢ ISO methods and other international standard methods	➢ ISO-TS 80004 – 2010 ➢ ISO-TS 12805 – 2011 ➢ ISO-TR 13014 – 2012 ➢ More than 30 National Standards	➢ As references from ISO, JIS, CNS and the ASTM, 52 National testing standards and testing methods have been established	➢ ISO methods and other international standard methods
6 Incentives/ facilitation provided	➢ Assistance for commercialization	➢ Facilitation for commercialization ➢ Business Training , ...	➢ Assistance for commercialization	➢ None

Source: nanoverify

Why Certification Mark (Labeling)

- Protect Consumers : Avoid waste money
- Protect Good Companies : Eliminate unfair competitions between good and bad products
- Increase Public Trust : Facilitate healthy development of nanotechnology
- Facilitate Trade : Stimulate economic growth

⇨ **Accelerate Good Nanoproduct Development**

Taiwan Nano Mark System

Introduction

History & Motivation

Policy

- National Nano Project development(2003)
- Legislators Requirements
- Directives of Ministry of Economic Affairs(2004)

Consumer

- To protect the interests of consumers
- To avoid confusion for consumers

Economic

- To encourage vendors' sustainable operation
- To enhance industry' competitiveness

Nano Mark Logo

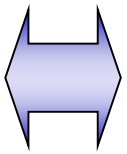
Implications:

1. **Round shape represents the feel of nano particle.**
2. **∞ symbolizes the unlimited application of nanotechnology.**
3. **Auxiliary English text expresses the verification and international link**
4. **Logo color will be set according to the product category.**

The Products Selection Criteria for Nano Mark

Product items criteria

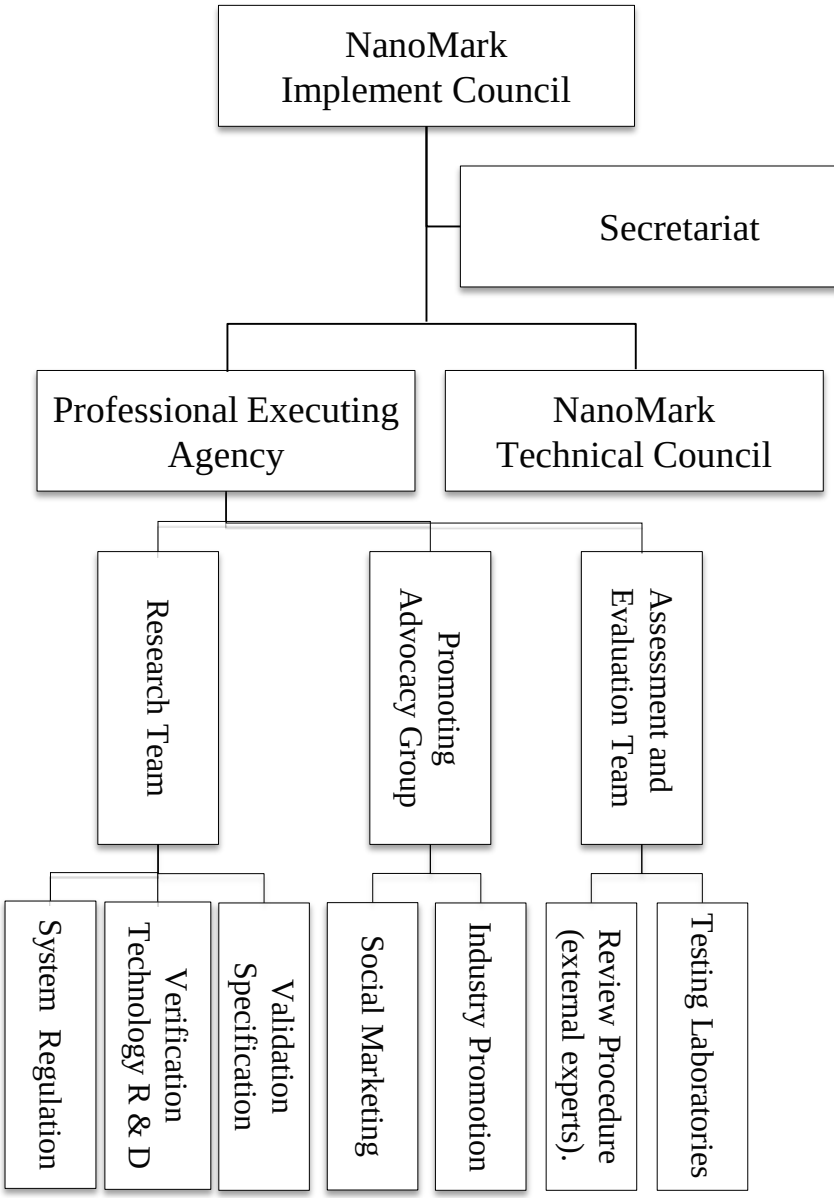
- Commercially available products, domestic manufactured products will be priority.
- Existing test method, testing laboratories, and technical experts.
- Products with obvious safety concern will not allowed for application.



Vendor criteria

1. Products possess nanometer size material and nano-enabled function.
2. Registered Company under the law of Taiwan.
3. Company with third party quality verification certificate.

Tasks of the Nano Mark System (2004-2016)



Tasks of NanoMark Implement Council
Executive board of nanoMark, system coordination, promotion, implementation issues.

Tasks of Secretariat
Staff operations, technical member appointment, managing the grant/terminate of nanoMark, managing operation of professional executing agencies and technical council.

Tasks of NanoMark Technical Council
Examining product items, specifications and test methods, managing testing laboratories application/registration, investigating technical abnormal event/other technical issues.

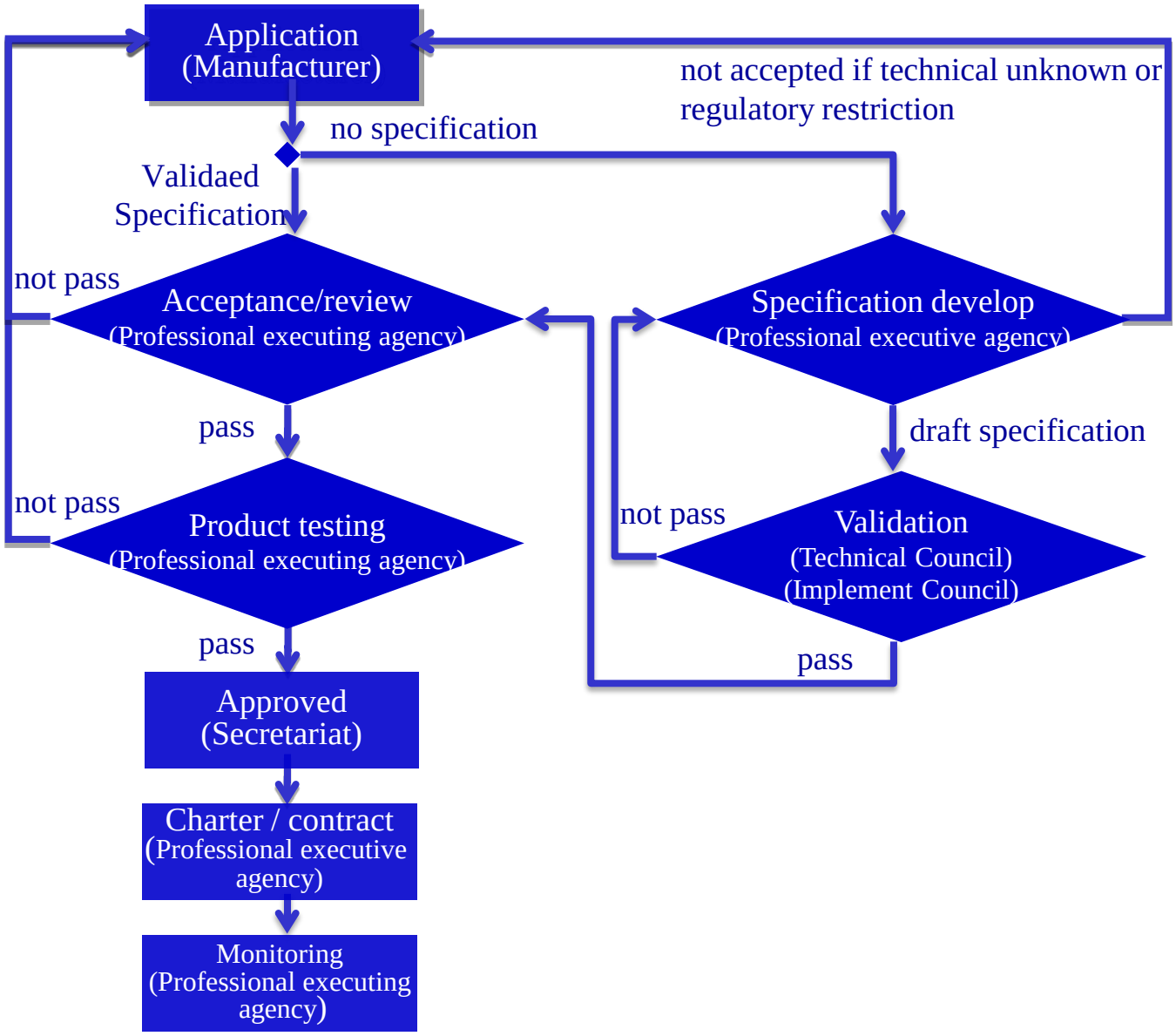
Tasks of Professional Executing Agency
Assessment and evaluation team: execution of vetting job

External expert members: application case review

The research team task: to develop validation and verify specification, research new areas for the appropriateness of acceptance, verify demands, legality and technical issues.

Promote advocacy group: promoting the work to community marketing, to promote the marketing to various industries.

Review/verification Process of nanoMark



Certified Products Examples

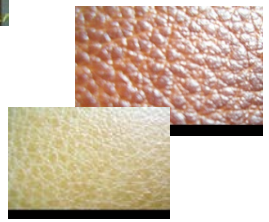
Textiles, Building Materials, Paint, Communication/Computer/ Consumer, Plastics & Rubber, Hardware/Metal, Others



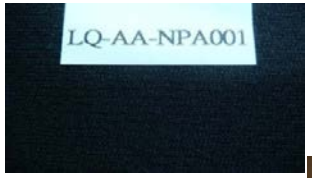


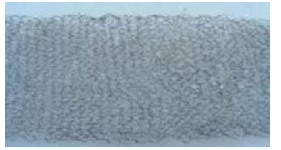








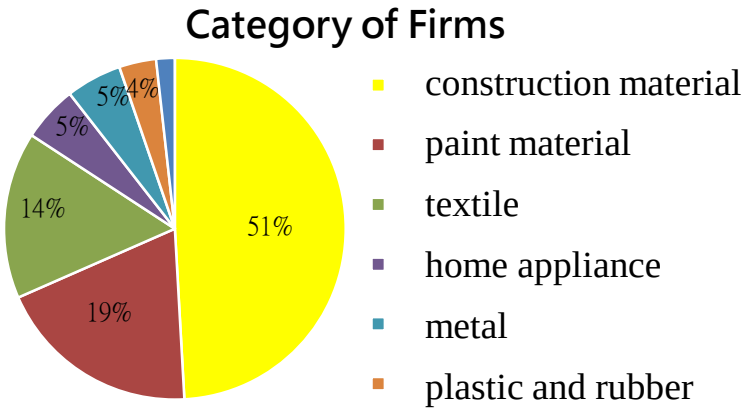


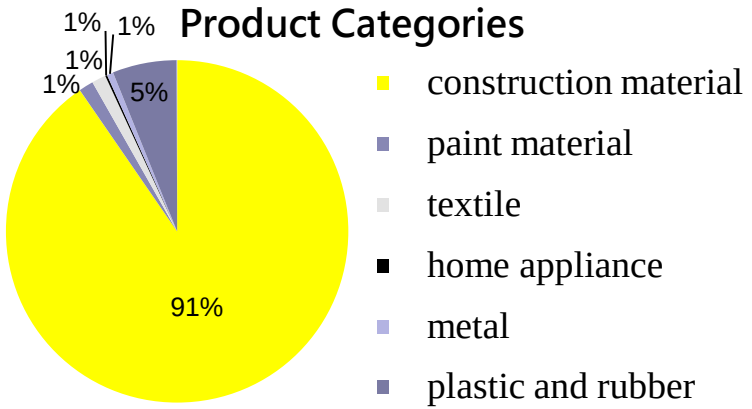

http://www.nanomark.org.tw/Eng/Product/

Current Condition of nanoMark

(Year 2004 to 2016)

- **52 published product certification standards**
- **55 total certified firms（2,715 products); 44 firms are still effective (2,454 products)**
  - 30 construction material firms （2232 products）
  - 12 paint material firms（30 products）
  - 8 textile firms（27 products）
  - 3 home appliance firms（14 products）
  - 3 metal firms（10 products）
  - 2 plastic and rubber firms（132 products）
  - 1 other firms（1 products）
- **14 Registered test laboratories（87 tests）**
  - 7 nano sizing
  - 5 anti bacterial
  - 10 others

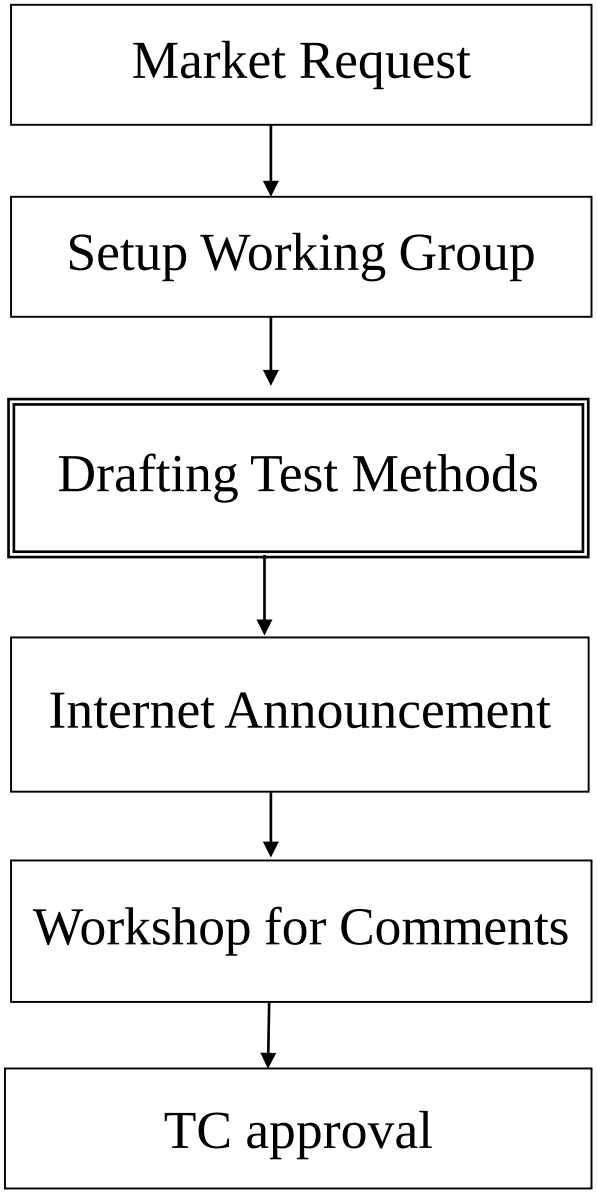




Taiwan Nano Mark System

Testing Standards

Procedure to Draft Test Methods for Nanoproducts



Select market preference of nanoproduct

Invite 3~5 experts to form a working group, feasibility discussion

1. collecting existing methods/standards
2. drafting one or more test standards
3. practicing draft standards
4. suggest criteria for NanoMark

Internet call for comments:
www.nanomark.itri.org.tw

Comments from targeted companies

TC discussion and approval, public announcement

Example(1)

photocatalyst (UV & Visible light) Anti-bacterial Tiles

- How many bacteria have to be tested ?
  - ✧ Representative
  - ✧ Safe／easy to handle

  ⇨ *Staphylococcus aureus*： ATCC 65389, BCRC 10451

  Escherichia Coli：ATCC 8739, BCRC 11634
- How effective anti-bacterial tiles should be ?
  - ✧ Achievable
  - ✧ Testing Protocol (conditions, raw materials and/or final products)

    ⇨ > 99%
  - ✧ Top 30~50% Companies
- What should be specified on the product package ?
  - ✧ How to use
    - ◆ Where
    - ◆ Who
    - ◆ How (application methods, maintenance, etc.)
  - ✧ Reliability

Example(1)

photocatalyst (UV & Visible light) Anti-bacterial Tiles

Tiles：N041 (900mm x 1800mm) (CERABO)

N04590 (450mm x 900mm) (Ohayo)

Who：Construction Companies, General Publics

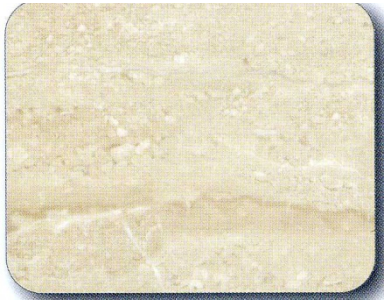
Where：Indoor (eg. toilet), Outdoor

How：• Adhesion to fix on the substrate

• Wet cloth to wipe if dirty

Reliability：real case (> 3 years)

Wall Tile (45 x 90 cm)



Floor Tile (90 x 90 cm)

Test：

Nano Scale：raw material, 40nm

Product, confirmed to have TiO_2

Anti-bacteria：*S. aureus* 99.99%

E. Coli 99.99%

365nm UV, 0.2mW/cm^2／24 hrs

S. aureus 99.99%

E. Coli 97.83%

543nm, 1000 lx／24 hrs

Quality System：ISO9001 (Certification：4XBY008-04)

Safety Issue：CNS 3298

Example(1)

– Photocatalyst (UV & Visible light) Anti-bacterial Tiles

Tiles：T627* (Champion Building Materials Co.)

Who : Construction Companies, General Publics

Where : Outdoor

How : • Adhesion to fix on the substrate

• Wet cloth to wipe if dirty or clean by rain

Reliability : > 5 years, company guarantee

Test：

Nano Scale：raw material, 32nm

Product, confirmed to have TiO_2

Anti-bacteria : *S. aureus* 99.99%

E. Coli 99.99%

365nm UV, 0.2mW/cm²／24 hrs

奈米標章 nano 004-00003

Quality System：ISO9001 (Certification：4XBY013-04)

Safety Issue：CNS 9742

抗菌陶瓷面磚

Example(2)

Anti-bacterial Socks

- How to define anti-bacteria without confusing consumer
  - ✧ de-odour purpose, not to cure skin diseases e.g. athlete's foot
- How many bacteria have to be tested
  - ✧ Representative
  - ✧ Safe/easy to handle
  - ✧ National standard (CNS 14945)

→ Staphylococcus aureus：ATCC 6538P

→ Klebsiella Pneumoniae：ATCC 4352

- How effective anti-bacteria socks should be (original, after wash)
  - ✧Achievable
  - ✧Test Protocol (conditions, raw materials and final products)
  - ✧Top 50% companies

→ *> 90%*

		Companies (local & abroad)					
Water Wash times	Anti-bacterial Socks	A	B	C	D	E	F
0	ATCC 6538P	99.8	99.9	< 0	> 99.9	> 99.9	< 0
	ATCC 4352	> 99.9	99.5	< 0	> 99.9	99.4	< 0
20	ATCC 6538P	> 99.9	98.8	--	97.3	80.2	--
	ATCC 4352	> 99.9	89.4	--	< 0	89.3	--

Example(2)
Anti-bacterial Socks

- **Safety Issues**
  - **What's the criteria**

    National Standard：CNS 14945
    - **Skin Irritation Test**
    - **Oral Acute Toxicity Test**

Anti-bacterial Socks	Company A
Skin Irritation	PII=0, no irritation
Oral Acute Toxicity	No abnormal phenomena or toxic response observed

- **Leaching Test**

One Sock in 6.8L H_2O/50℃/180rpm/12min

↓

Water sample

Atomic Absorption Analysis

↓

Ag < 0.01 ppm

(Taiwan EPA regulation：drinking water, Ag < 0.05 ppm)

Example(3)
Hydrophobic Car Wax

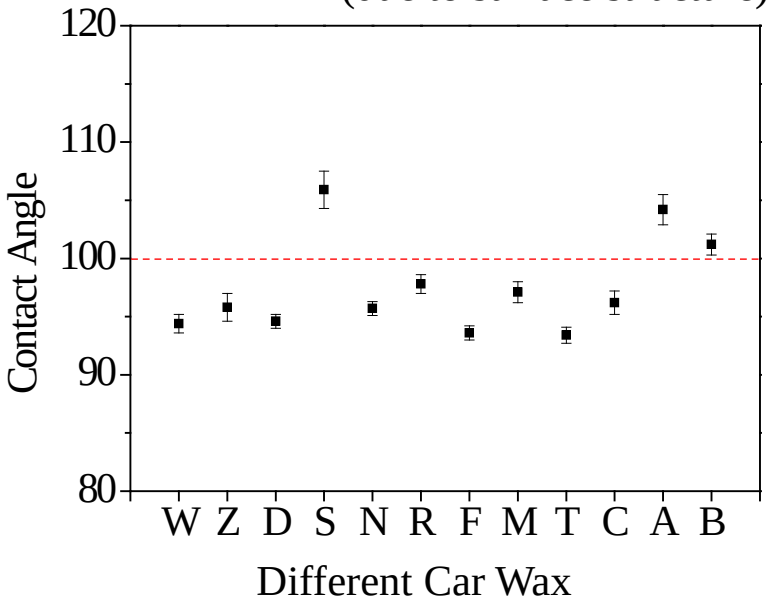
How to define "hydrophobic"?

Is "contact angle" enough?

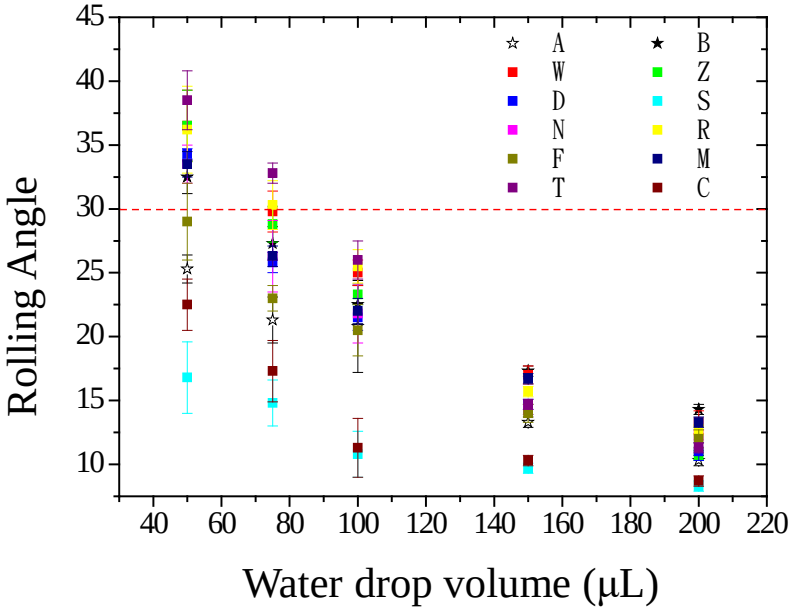
How high the contact angle should be?

16 Performance Tests needed in ASTMD 3836-94 (reapproved1999)

- Contact Angle：4μL Water drop, CA>100°
  (due to surface structure)



- Water drop rolling angle
  --50 μL water drop, rolling angle < 30°



Example(3)
Hydrophobic Car Wax

- Water blot area <40%

Water blot area at sliding angle 35º (50 mL/min)

5 : 0~20%, 4: 21~40%, 3 : 41~60%, 2 : 61~80%, 1 : >81%

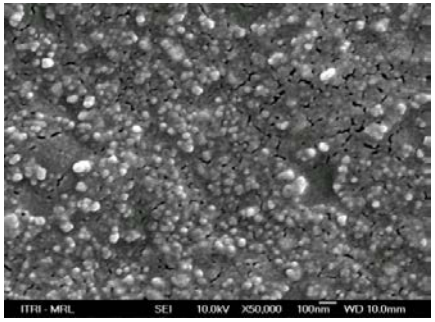
- Reliability：anti-washing (ASTMD 2486)
  > 1000 times, CA > 100°

Different Car Wax		
	A	5
	B	4
	W	3
	Z	3
	D	4
	S	5
	N	3
	R	4
	F	3
	M	2
	T	2
	C	5

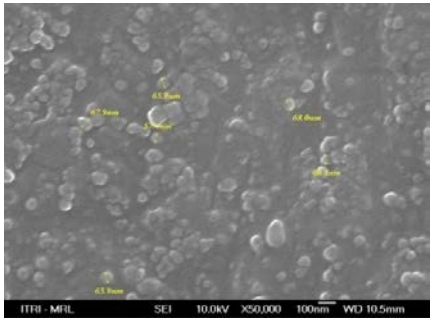
CA / Washing times		Brand A	Brand B
Tap water	0	104.2±0.9	103.6±0.3
	1000	104.8±1.0	104.0±0.6
With detergent	0	105.5±0.2	102.4±1.0
	1000	102.4±0.4	101.0±5

- Nanoscale：nanostructure revealed on car body surface after waxing

SEM Analysis



Brand A Wax

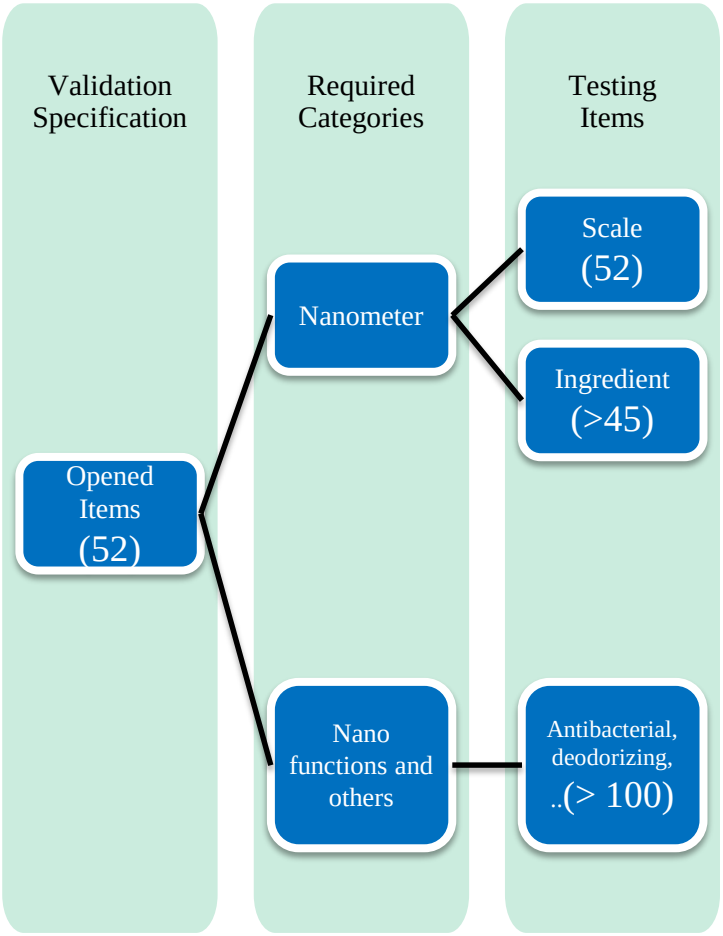


Brand B Wax

Taiwan Nano Mark System

Testing Laboratory

Testing services of the registered laboratories



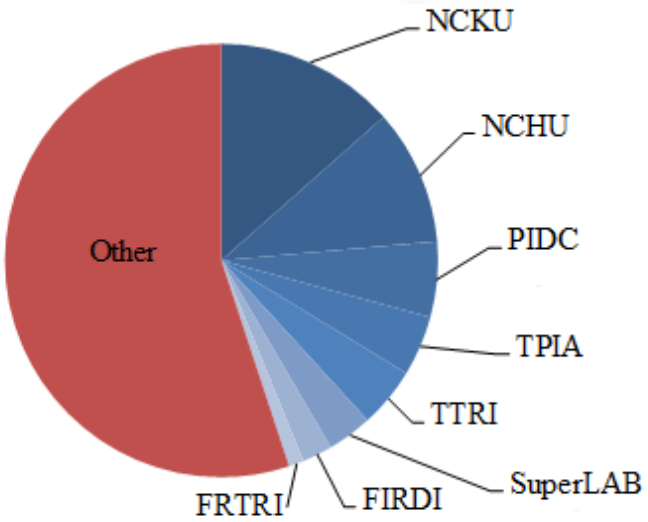
Total of 14 registered laboratory ~ 200 test items

Nanoscale coverage rate of registered labs 100%

SEM	Nano Facility Center、NTHU、NCKU
TEM	Nano Facility Center、NCHU、MSSCORPS.CO
AFM	Nano Facility Center、LHU
DLS	NCHU、NCKU
XRD	NCHU
EDS	(With nanoscale instrumentation)

Nano function coverage rate of registered labs ~50%

Test items distribution in each laboratory



SEM: Scanning Electron Microscopy
TEM: Transmission Electron Microscopy
AFM: Atomic-Force Microscopy
DLS: Dynamic Light Scattering
XRD: X-Ray Diffraction
EDS: Energy-Dispersive Spectrometry

Bioresource Collection and Research Center, Food Industry Research and Development Institute (Lab. Code：0001)

No.	Test Item	Test Method
01	Antibacterial rate of staphylococcus aureus and E. coli	JIS Z2801

Common Nano Facility Laborotory, Industrial Technology Research Institute (Lab. Code：0004)

No.	Test Item	Test Method
01	Nanoscale	SEM, TEM, SPM protocols

Center for Micro/Nano Science and Technology, National Cheng Kung University (Lab. Code：0006)

No.	Test Item	Test Method
01	Nanoscale powder	DWI-T-S1(SEM) (10 nm to 700 nm) ISO 13321(DLS) (20 nm to 1000 nm)
02	Antibacterial rate of klebsiella pneumoniae & staphylococcus aureus	JIS Z2801、ISO 22196
03	Skin irritation	Rabbit skin irritation test inspection procedures - textiles, cosmetics, medical equipment and chemical reagents
04	Acute oral toxicity	Acute oral toxicity test (limit method) inspection procedures - medical equipment, cosmetics, food, chemicals and textiles

Nano-material Measurement Laboratory, National Chung Hsing University (Lab. Code：0007)

No.	Test Item	Test Method
01	Nanoscale	DLS, TEM
02	Antibacterial rate of staphylococcus aureus and E. coli	JIS Z2801、ISO 22196
03	Deodorization rate (acetaldehyde)	TN-001
04	Deodorization test	TN-009、TN-010
05	Contact angle	TN-008、TN-12
06	Residual contaminants	TN-008、TN-12
07	Decomposition test of methylene blue	TN-031

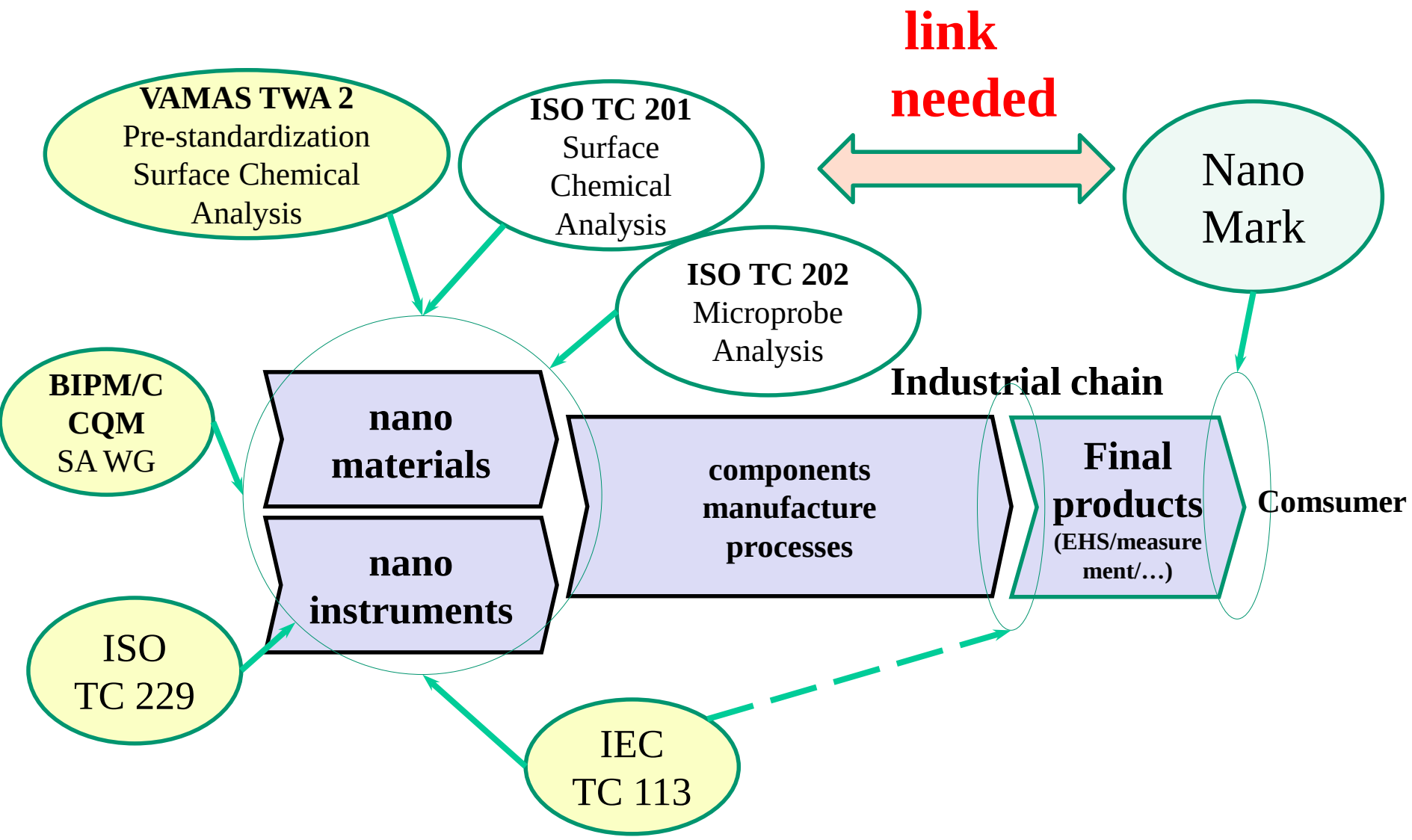
Certification and test laboratory, Footwear & Recreation Technology Research Institute (Lab. Code：0009)

No.	Test Item	Test Method
01	Abrasion resistance	CNS 12915

Research and Development Laboratory, Taiwan Paint Industry Association (Lab. Code：0012)

No.	Test Item	Test Method
01	Adhesion	CNS10757
02	hardness	CNS10757
03	Scrub resistance test	CNS10757
04	Gloss test	CNS10756
05	Abrasion test	CNS10757
06	Salt spray test	CNS11607
07	QUV weathering test	CNS12147 QUV
08	Alkali test	CNS10757

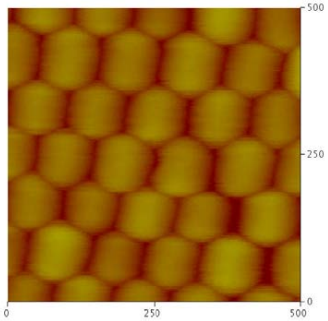
Standards activities involved



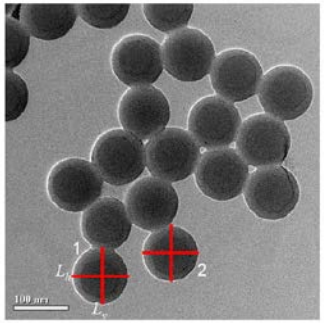
Early testing laboratories comparison

Example testing laboratory measurement

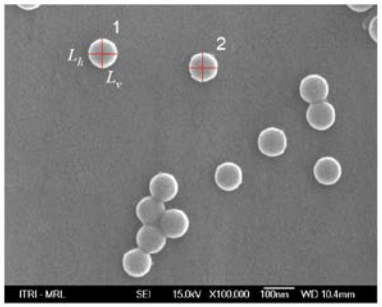
SPM

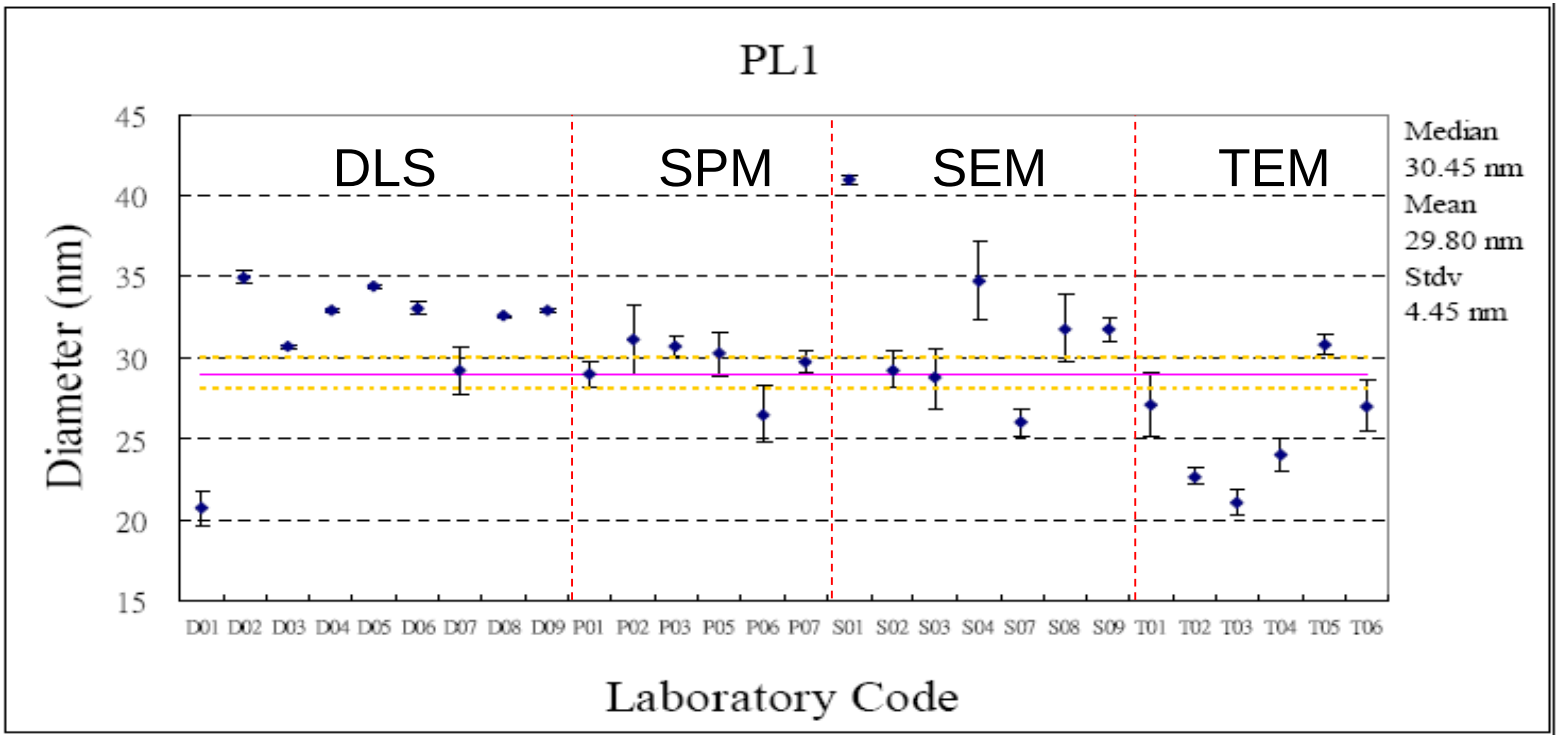


SEM



TEM





Inter-laboratory comparison

1st example: APEC Nano product measurement comparison

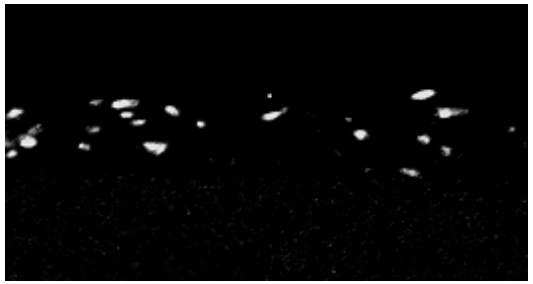
Selected comparison products: self cleaning glass

Nano characteristics

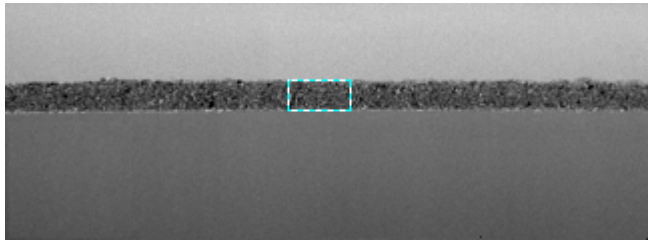
The self cleaning composition and size measurement of photo catalysis glass

- Test Instrument: TEM (dark field) or others

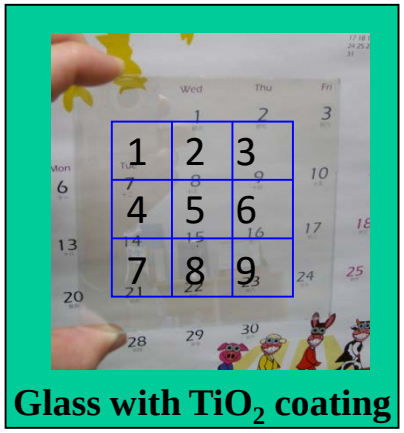
Sample preparation with FIB



Size distribution (Dark field results)



TiO_2 thickness

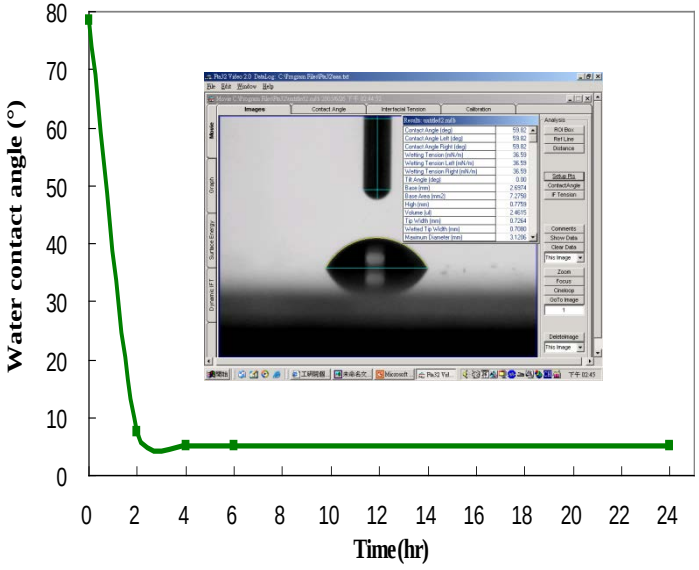


Glass with TiO_2 coating

Functional characteristics

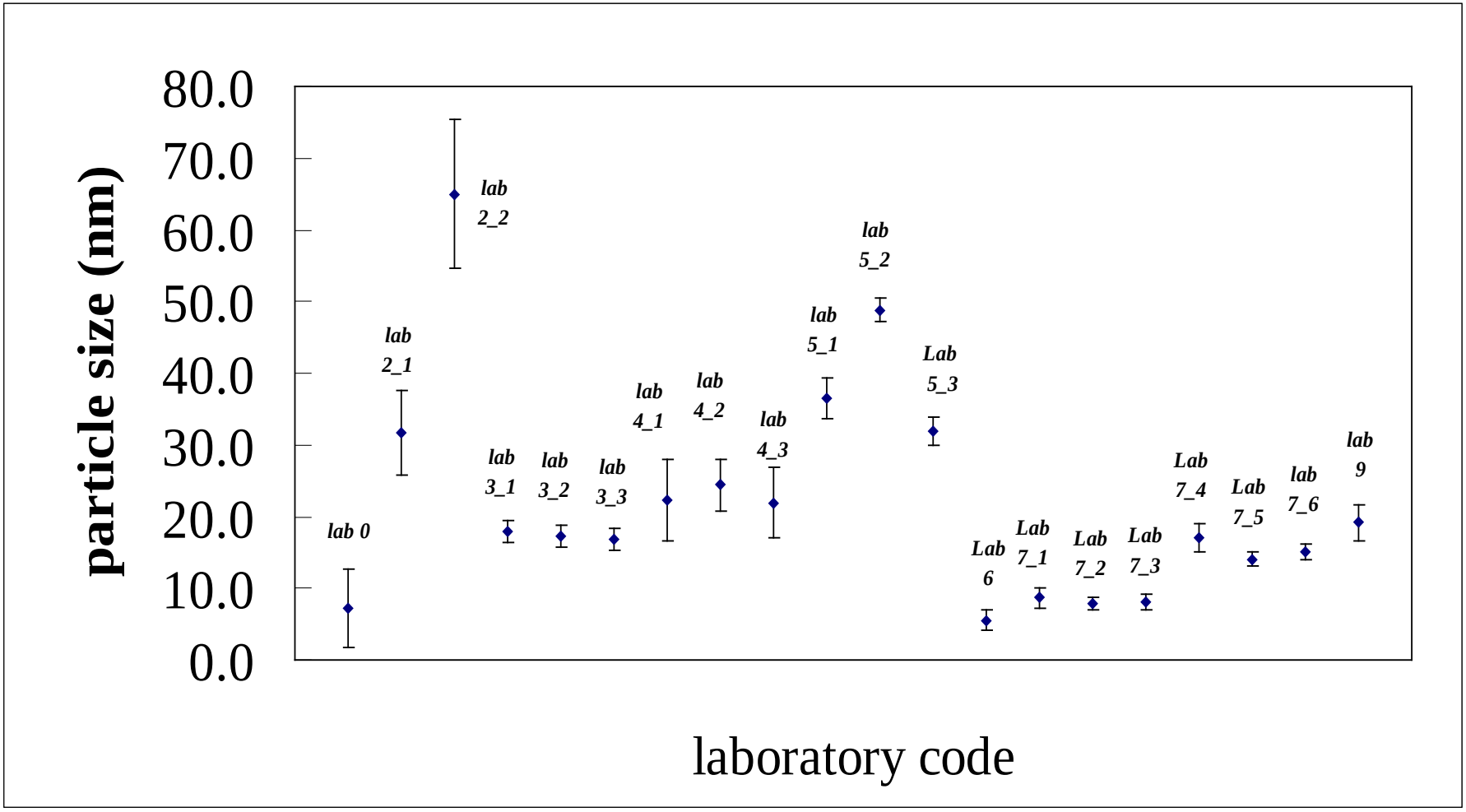
The self cleaning efficiency measurement of photo catalysis glass

- **Glasses：B270-Superwite, 2 mm thickness**
- **Test method: similar to JIS R 1703-1**
- **TiO_2 type：Anatase**
- **Surface coating of oil or acid: immersion plating**
- **Self cleaning efficiency : contact angle <10°（24 h)**



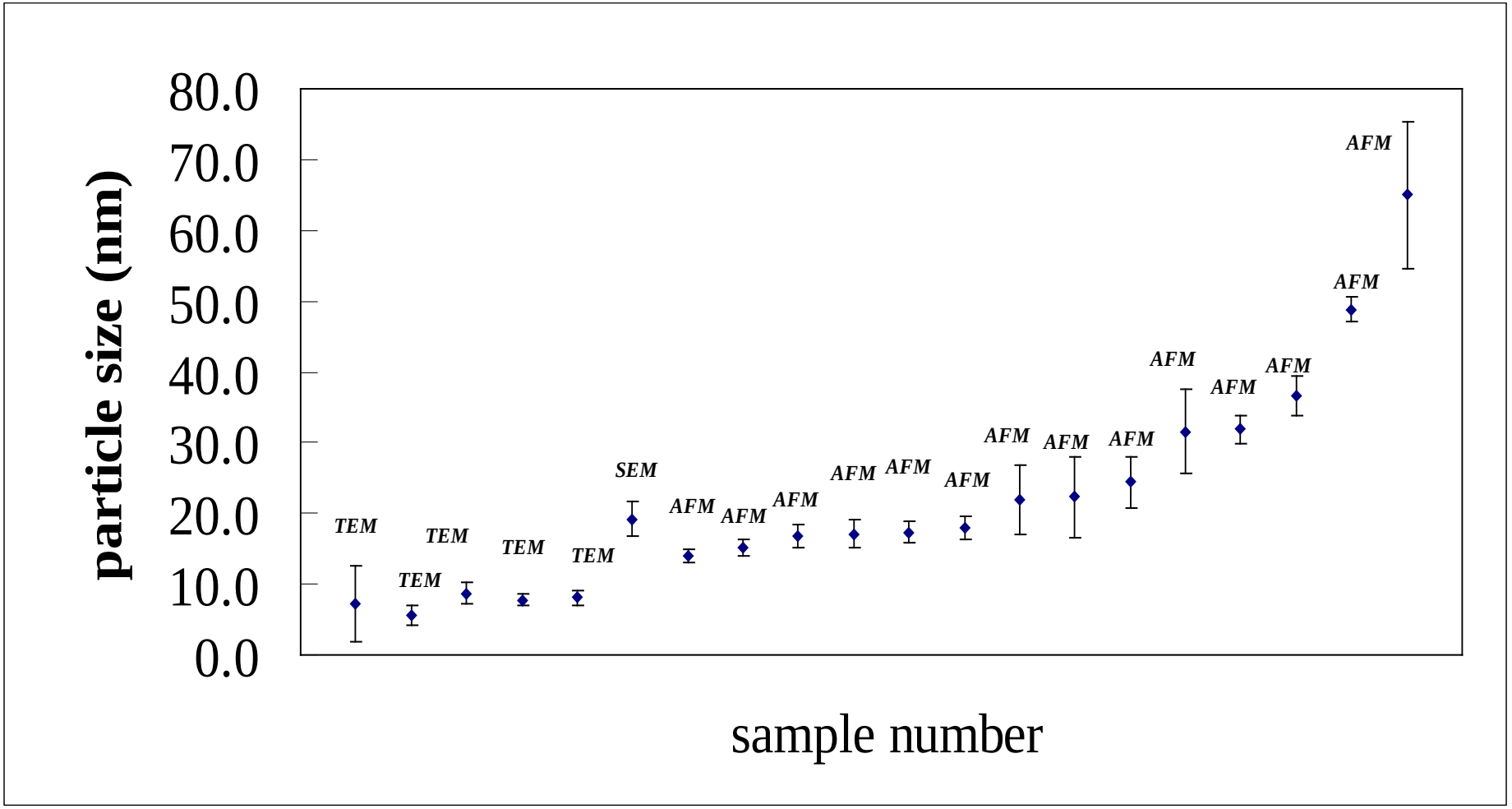
How different is the data between labs. ?

Nano characteristics(particle size) from nano product

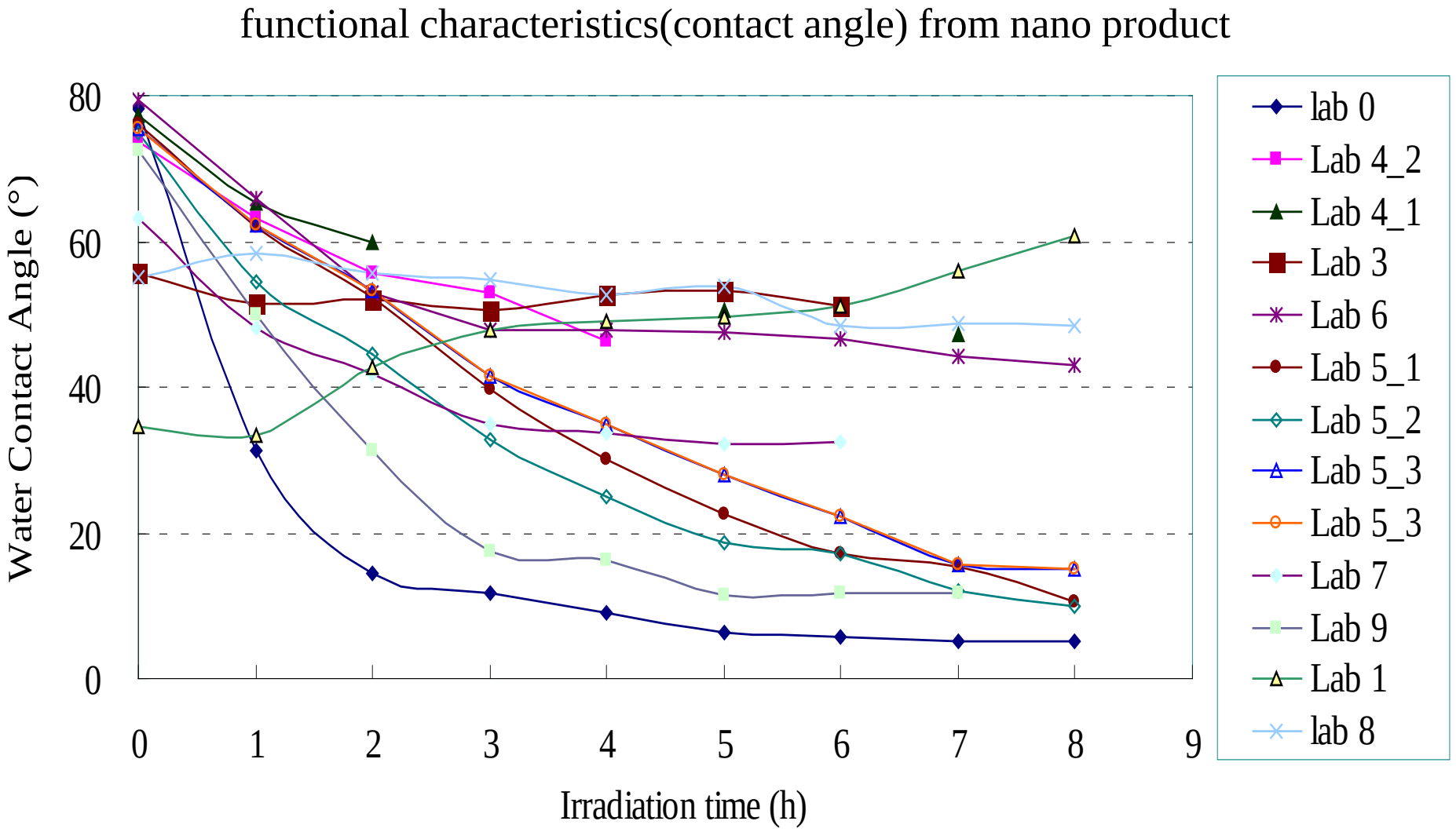


How different is the data between labs. ?

Nano characteristics(particle size) from nano product



How different is the data between labs. ?



Recent testing laboratories comparison

Participants and Nanoparticles

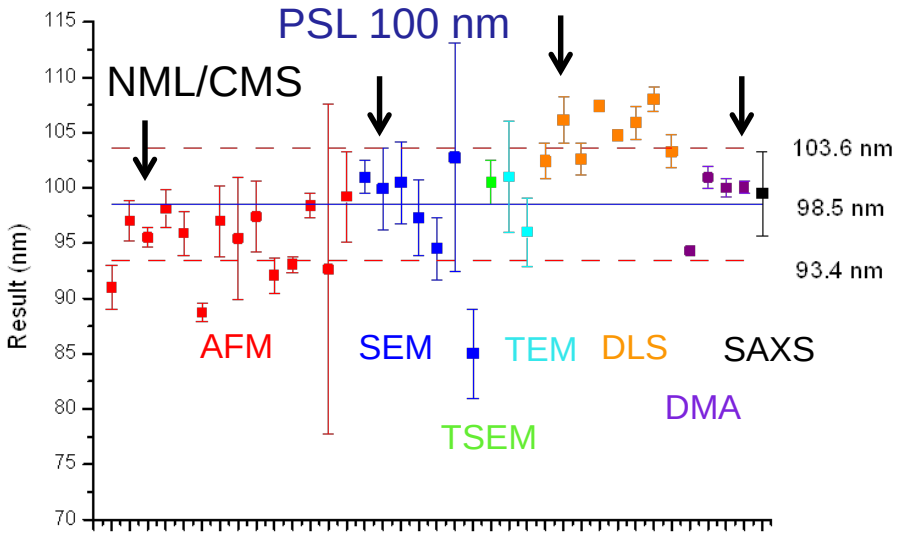
APMP.L-S5,
Length, Dimensional Metrology, Nano particles measurements
Joint Comparison with TCMM in APMP (draft B report publishing soon)

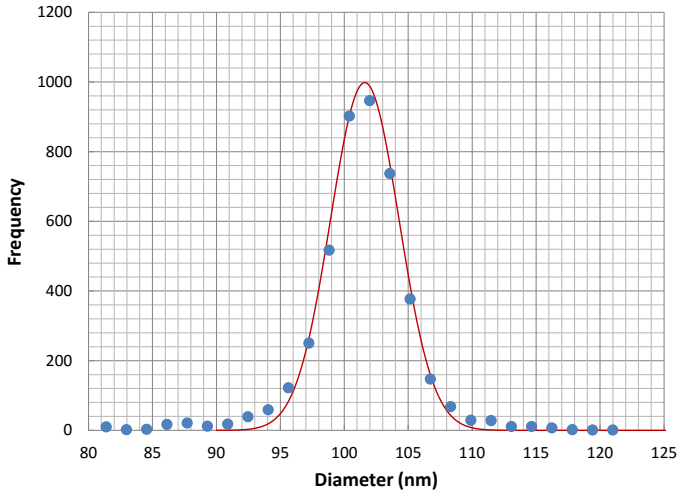
Pilot laboratories	**CMS/ITRI, NMIJ**
Participants	**CENAM, CMS, DFM, INMETRO, INRIM, KRISS, LNE, METAS, NIM, NIMT, NMIA, NMISA, NMIJ, PTB**
Methods	**AFM, SEM, TEM, DLS, DMA, XRD, SAXS**

No.	Material	Nominal size [nm]	Volume [mL]	Number concentration [particles/mL][1]	Manufactur
G1	Nano Gold	10	2	5.7×10^{12}	BBInternatic
S2	Nano Silver	20	2	4.0×10^{11}	nanoCompo
P3	Polystyrene latex	30	1	7.0×10^{14}	JSR
P4	Polystyrene latex	100	1	1.8×10^{13}	JSR
P5	Polystyrene latex	300	1	7.1×10^{11}	JSR

Measurement Results

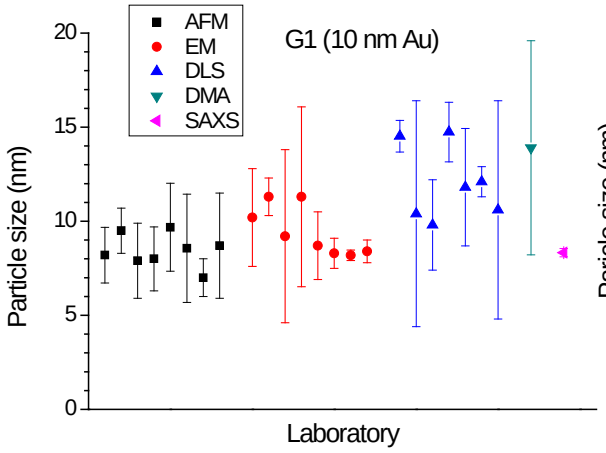
Materials *	Gold 10 nm	Silver 20 nm	PSL** 30 nm	PSL 100 nm	PSL 300 nm
AFM	8	8	10	14	12
EM	8	8	4	10	9
DLS	8	8	8	8	8
DMA	2	0	3	4	4
SAXS	1	1	1	1	1

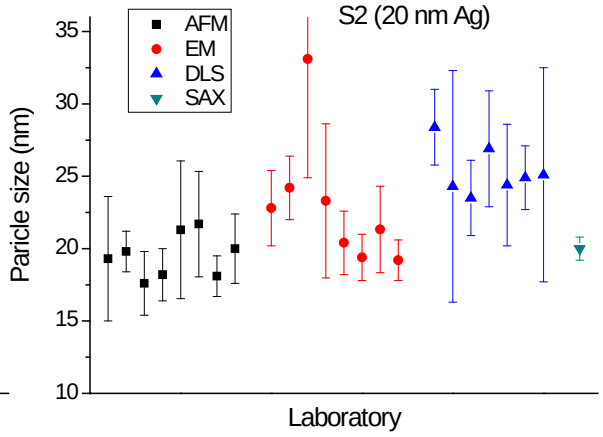


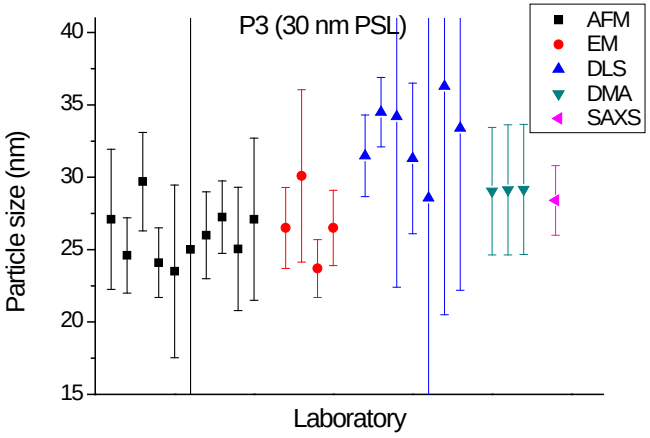


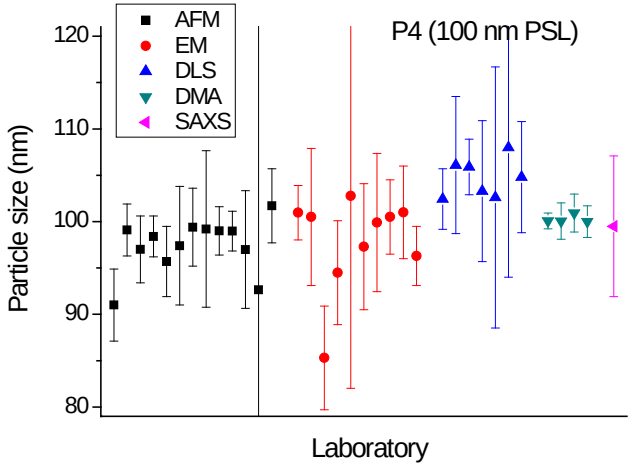
P4 particles obtained by the TSEM at PTB (solid circles), and the Gaussian distribution fitted to the experimental data (solid curve).

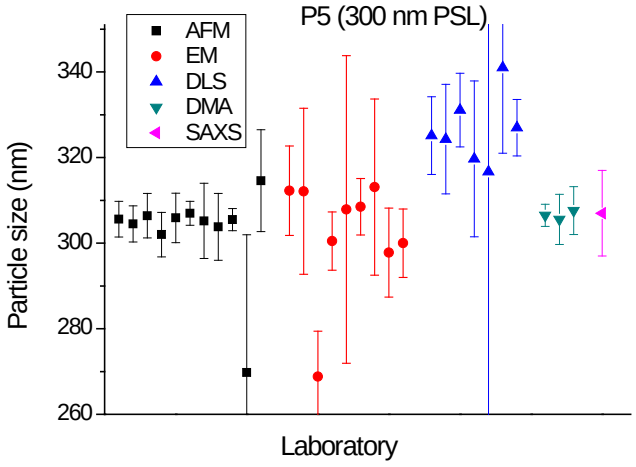
Whole data reported in Draft A2





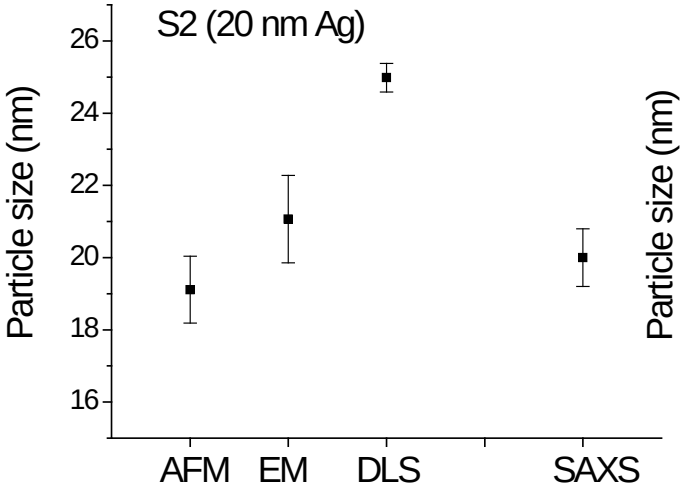


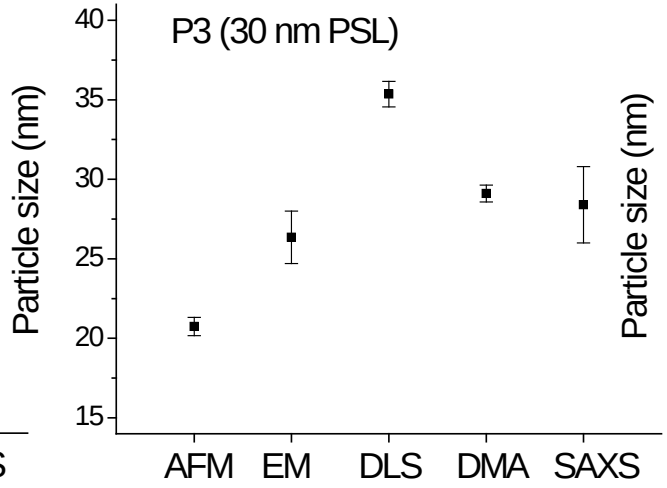


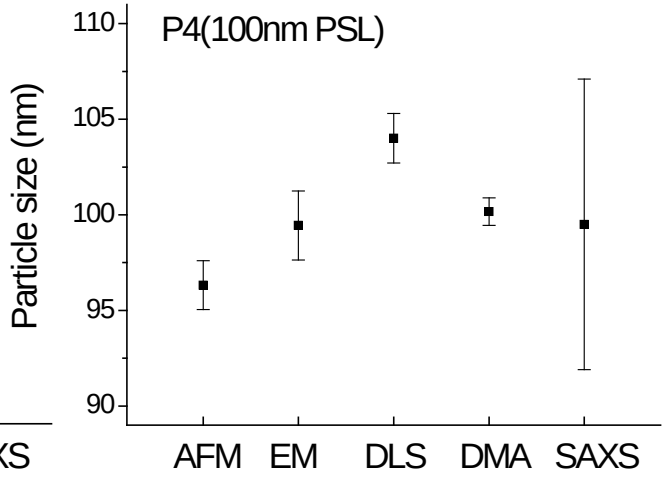


Draft A indicated that:

- AFM tends to give smaller particle sizes than the other methods
- DLS tends to give larger particle sizes than the other methods

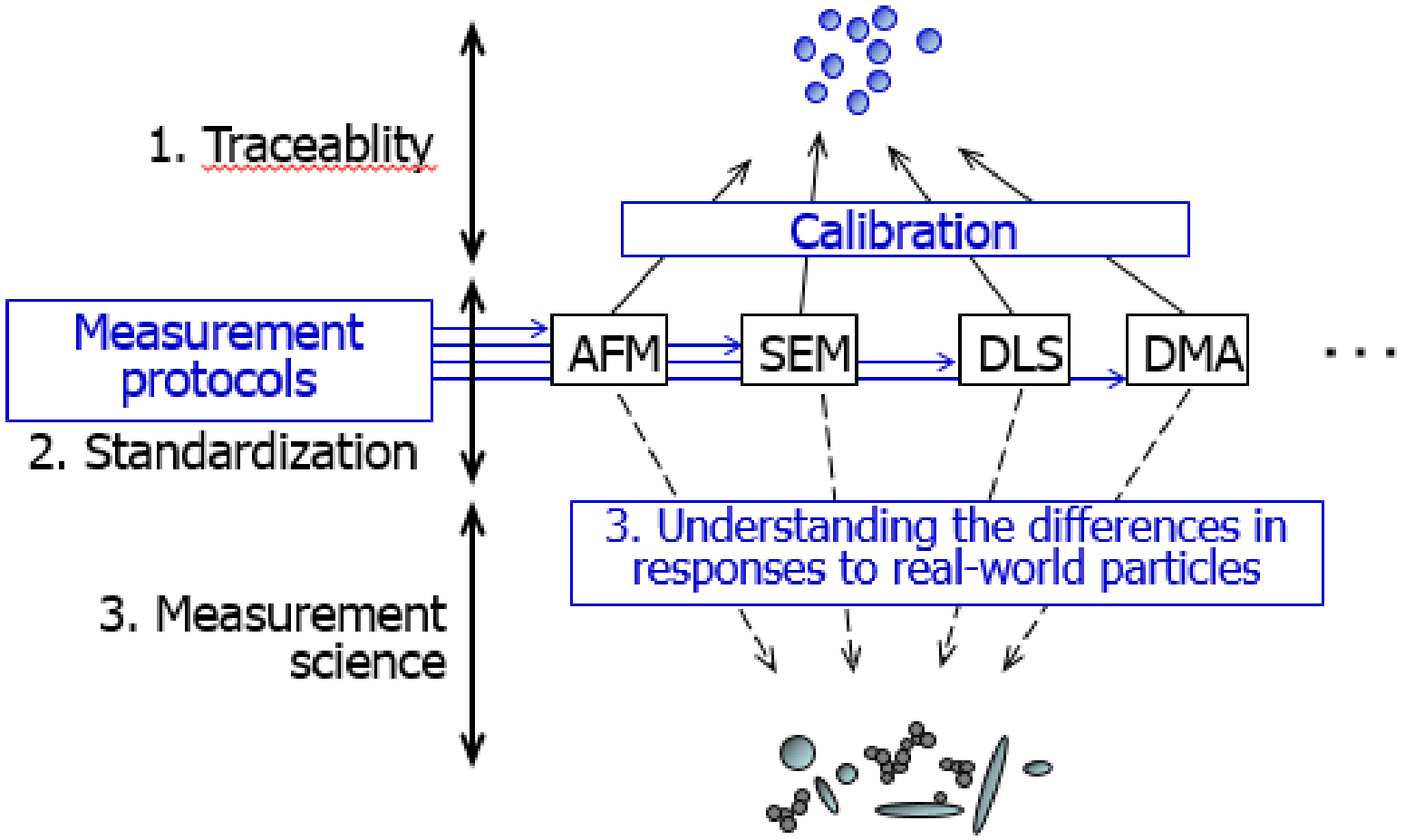






- Globe Reference Values include all methods
- Method Dependent Reference Values

Scheme to establish harmonized measurement infrastructure



- Different measurands for different measurement methods will not help establish a harmonized measurement infrastructure for nanoparticles

Some Summaries from Workshops

1. #Definition of the measurand (from 2014):
   We intend to measure the core diameter of nanoparticles. The measurand is the "mean equivalent diameter of particles of a specific material."
   This draws on the definition of equivalent diameter from ISO TS 80004-6:2008, clause 3.1.5 and on particle from ISO TS27687:2008, clause 3.1. We think that it is important to include 'equivalent' in the description, as we make the assumption that the particles are spherical. We also feel that it is important to make clear the distinction that we are measuring the mean diameter of a population of particles, not just a single particle.
2. #Definition of terms (from 2015):
   We decided to use two terms 'method-dependent reference value' and 'global reference value.'

Acknowledgements above comparison slides

- The slides for the data analysis are referred to the presentation slides from Dr. K. Ehara (NMIJ) and Ms. H.L. Lin (CMS).
- The data analysis for technical parts are provided from the Colleagues in CMS/ITRI, colleagues in NMIJ, all participants in APMP.L-S5 and Dr. Dagata's group in NIST.

Outlook

Global Collaborations → Consistent Criteria

→ Test lab. Recognitions → Mutual Recognitions

Taiwan	Iran	Thailand	Malaysia
2004	2009	2011	2015
Voluntary	Mandatory if apply government support	Voluntary	Voluntary

→ Accelerate Good Nanoproduct Development

Thanks for Your Attention !